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STUDY AND EVALUATION OF SEVERAL SMOKE-GENERATING MATERIALS FOR --ETC(U)  
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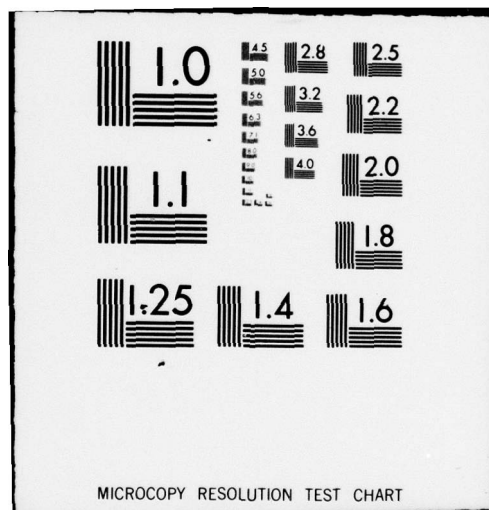
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STUDY AND EVALUATION  
OF SEVERAL SMOKE-GENERATING MATERIALS  
FOR USE IN HAZARD-FREE CARTRIDGES  
FOR 81-mm MORTAR INERT TRAINING AMMUNITION

BY

Alcuin F. Gremillion  
Daniel A. Lieblong

BEI Electronics, Inc.  
1101 McAlmont Street  
PO Box 3838  
Little Rock, Arkansas 72203

15 January 1979

Final Report

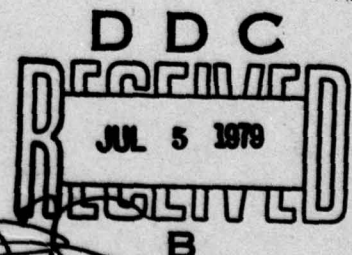
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field tests in 81-mm mortar training rounds. The two that are most preferred are chemical mixtures that produce smoke spontaneously and instantly when the components are brought together. The third is a slow-burning mixture. An assessment of toxic hazards from the chemicals used is included.

## SUMMARY

Seven smoke-producing systems were used in the laboratory to produce smokes or dusts under conditions which would enable comparison of the products from the standpoint of quality and quantity of smoke and ease of production.

The major portion of the data is presented in the form of photographs.

Three of the systems studied are recommended as candidates for field tests in 81-mm mortar training rounds. The two that are most preferred are chemical mixtures that produce smoke spontaneously and instantly when the components are brought together. The third is a slow-burning mixture.

An assessment of toxic hazards from the chemicals used is included.

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## CONTENTS

	Page
ILLUSTRATIONS	iv
TABLES	vii
INTRODUCTION	1
EXPERIMENTS	3
1. Apparatus and Procedure	3
A. Procedure with Spontaneously Reacting Mixtures	4
B. Procedure for Dispersing Day-Glo Orange Powder	6
C. Procedure for Burning LTSB Mixtures	12
2. Chemicals and Chemical Mixtures	15
3. Photographs and Other Data Forms	17
4. Experimental Results and Discussion	18
A. Spontaneous Reaction between Sulfuryl Chloride and Aqueous Ammonia - Experiments 1, 2, 3, and 4	18
B. Spontaneous Reaction between Acetyl Chloride and Aqueous Ammonia - Experiments 5, 6, and 7	24
C. Spontaneous Reaction Between Titanium Tetrachloride and Water - Experiments 8, 9, 10, and 11	28
D. Dispersion of Day-Glo Orange Powder With Air - Experiments 12 and 21	35
E. Smoke Generation from LTSB Mixture I - Experiments 13 and 14	42
F. Smoke Generation from LTSB Mixture III - Experiments 15, 16, and 17	49
G. Smoke Generation from LTSB Mixture II - Experiments 18, 19, and 20	50
5. Comments on the LTSB Mixtures	56
6. Properties of Chemicals Used	58
RECOMMENDATIONS	62
DESIGN AND COST	63
1. Cost Estimate	65
2. Attachment I	66
3. Attachment II	67
4. Attachment III	68
REFERENCES	69



## ILLUSTRATIONS

Figure	Title	Page
1.	Apparatus for Generating Smokes from Spontaneously Reacting Chemical Mixtures	5
2.	Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 before Introduction of Air into the Chamber	8
3.	Glass Apparatus Arrangement Through Which Day-Glo Orange Powder was Introduced into Dispersion Chamber by the First Method	9
4.	Glass Apparatus Arrangement With Which Day-Glo Orange Powder was Dispersed by the Second Method	10
5.	Apparatus used in Dispersion of Day-Glo Orange Powder by the Second Method in Experiment 21	11
6.	Apparatus for Generation of Smokes from LTSB Mixtures I and III in Experiments 13, 14, 15, 16, and 17	13
7.	Smoke at Conclusion of Burning LTSB Mixture II in Experiment 19	14
8.	Apparatus for Burning LTSB Mixture II in Experiments 19 and 20	16
9.	Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 1	20
10.	Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 2	21
11.	Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 3	22
12.	Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 4	23
13.	Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 5	25
14.	Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 6	26
15.	Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 7	27



# ILLUSTRATIONS (contd)

Figure	Title	Page
16.	Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 8	30
17.	Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 9	31
18.	Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 10	32
19.	Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 11	33
20.	Smoke from Reaction of Titanium Tetrachloride and Aqueous Ammonia in Experiment 22	36
21.	Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 while Introducing Air into the Chamber after Day-Glo Orange Introduction	37
22.	Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 after Bringing Pressure inside the Chamber up to One Atmosphere	38
23.	Dispersion of Day-Glo Orange Powder by the Second Method in Experiment 21	41
24.	Smoke at an Early Stage of Burning LTSB Mixture I in Experiment 13	43
25.	Smoke at a Late Stage of Burning LTSB Mixture I in Experiment 13	44
26.	Smoke at Conclusion of Burning LTSB Mixture I in Experiment 13	45
27.	Smoke at an Early Stage of Burning LTSB Mixture I in Experiment 14	46
28.	Smoke at a Late Stage of Burning LTSB Mixture I in Experiment 14	47
29.	Smoke at Conclusion of Burning LTSB Mixture I in Experiment 14	48
30.	Smoke at an Early Stage of Burning LTSB Mixture III in Experiment 15	51

## ILLUSTRATIONS (contd)

Figure	Title	Page
31.	Smoke at Conclusion of Burning LTSB Mixture III in Experiment 15	52
32.	Smoke at an Early Stage of Burning LTSB Mixture III in Experiment 16	53
33.	Smoke at Conclusion of Burning LTSB Mixture III in Experiment 16	54
34.	Smoke at Conclusion of Burning LTSB Mixture III in Experiment 17	55
35.	Smoke at Conclusion of Burning LTSB Mixture II in Experiment 20	57
36.	Practice Fuze, 60/81-mm Mortar Hazard-Free, Binary Chemical Smoke	64

## TABLES

Table	Title	Page
I.	LTSB Mixtures Compositions	17
II.	Conditions Employed in Experiments 1, 2, 3, and 4. Reacting Sulfuryl Chloride and Aqueous Ammonia	19
III.	Conditions Employed in Experiments 5, 6, and 7. Reacting Acetyl Chloride and Aqueous Ammonia	28
IV.	Conditions Employed in Experiments 8, 9, 10, and 11. Reacting Titanium Tetrachloride and Water	29



## INTRODUCTION

On behalf of BEI Electronics, Inc., limited experiments were carried out by the Chemistry Department of the University of Arkansas at Little Rock. The following is a report of the results of these experiments.

For the most part, each experiment consisted of generating a smoke or chemical cloud within an enclosure and the photographic recording of the cloud produced, so the results could be compared visually.

The experiments were conducted in accordance with the constraints imposed by the space available for a smoke cartridge within an 81-mm mortar round. Quantity and quality of smoke produced were compared. The physical and toxicological hazard of each component of each generation mixture has been assessed by consulting readily available literature on physiological activity and other pertinent properties of the components.

Smoke was generated by three means:

1. by spontaneous reaction upon mixing two chemicals where the reaction is not one of burning,
2. by forced dispersion of a powder by some physical force, and
3. by heat activation of a low-temperature, slow-burning (LTSB) "cool" smoke mixture.

The seven smoke-producing systems are as follows:

1. Spontaneous Chemical Reaction
  - a. Aqueous ammonia (ammonium hydroxide) and sulfuryl chloride.
  - b. Aqueous ammonia and acetyl chloride.
  - c. Titanium tetrachloride and water.
2. Forced Dispersion of A Powder

Air dispersion of Day-Glo Orange powder.



3. Heat Activation of LTSB Mixture

- a. LTSB Mixture I, a formulation consisting of sulfur, sodium bicarbonate, potassium chlorate, and Uvinul 400 dye.
- b. LTSB Mixture II, a formulation consisting of zinc oxide, hexachloroethane, and aluminum powder.
- c. LTSB Mixture III, a formulation consisting of 1,4,5,8-tetrachloroanthraquinone, potassium chlorate, and confectionary sugar.

The choice of materials and mixtures, and the details of experimental procedure were dictated by considerations of time and availability of materials and apparatus. This is discussed in the following paragraphs where appropriate.

## EXPERIMENTS

### 1. Apparatus and Procedure

The space available for materials in the 81-mm mortar training round fuze cavity was computed at 50 ml. Accordingly, it is assumed that no more than 40 ml would be available for chemicals. This constraint is the basis for the amount of chemicals used in the experiments. Total volume of chemicals used in each experiment did not exceed about 4 ml, based upon a planned reduction by one order of magnitude.

In order to achieve good visibility, reproducible reaction and dispersion vessel volume, rapid removal of vapors and fumes, and rapid mixing in the case of spontaneous reactions, a 2l-liter round-bottom Pyrex glass flask housed in a fume hood was chosen, as shown in the following photographs. In most of the experiments where mixing of spontaneously reacting chemicals and burning of LTSB mixtures were conducted, the pressure in the flask before mixing or ignition was less than 1 mm Hg.

All photographs were taken under existing lighting conditions, i.e., the lights that were built into the fume hood. The camera was a 35-mm Pentax Spotmatic with 50-mm lens. The average exposure was f/2.8 at 1/15 s. The film was EPD 404. The film was developed by the standard E-6 process.

#### A. Procedure with Spontaneously Reacting Mixtures

Figure 1 shows the experimental arrangement used for spontaneously reacting chemicals. Each chemical was weighed into its introduction chamber. These are glass tubular chambers fitted with stopcocks and attached to the 2l-liter flask through standard taper joints at the top and left side of the flask. The chemicals were placed into position only after the pressure inside the flask was reduced below 1 mm Hg. Each chemical was isolated from the atmosphere by a stopcock at the top of the chamber.

As soon as the chemicals were in position, the stopcock between the flask and the vacuum line was closed. One of the chemicals was immediately introduced into the flask where much, if not all of it, vaporized. The stopcock to the first introduction chamber was closed and the second chemical was then introduced. In all of these cases there was instantaneous reaction and development of a smoke throughout the flask. Several photographs were taken in rapid succession as soon as the second chemical was introduced. This was done until long after no visible change inside the flask could be observed. In no case did the period of visible change extend beyond about five seconds, and in the last second or two of this period, changes were minor.

Several preliminary experiments with sulfuryl chloride and ammonia were conducted in which a movie camera was employed. The results from these were compared with results from experiments in which the 35-mm camera was used. The development of smoke was so rapid that



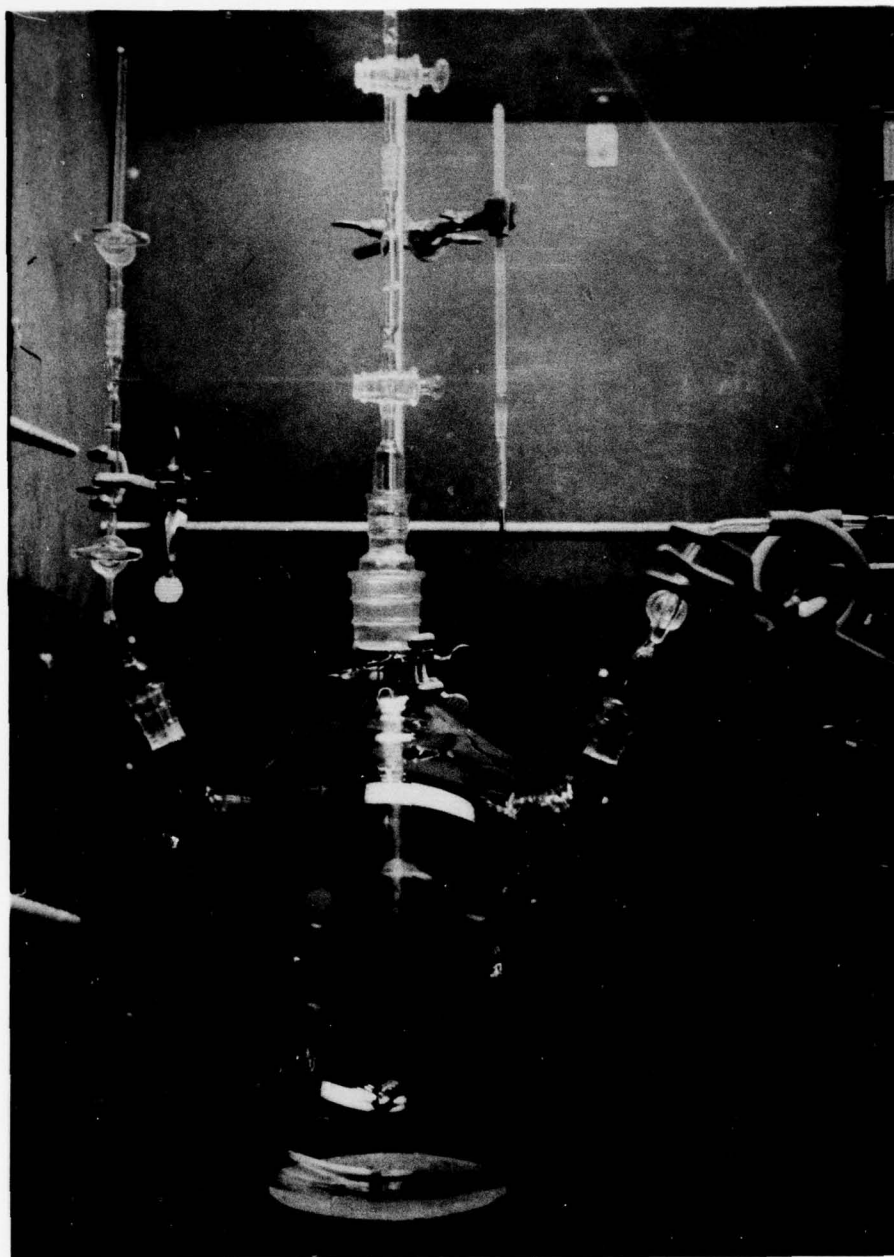


Figure 1. Apparatus for Generating Smokes from Spontaneously Reacting Chemical Mixtures



the framing rate of a standard movie camera offered no substantial advantage over the still camera.

B. Procedure for Dispersing Day-Glo Orange Powder

Inspection of the Day-Glo Orange powder as received showed that while it consists of a finely divided solid, some condition of this material produces substantial cohesion of particles. This is shown by the strong tendency toward formation of soft lumps. As these lumps were broken up with a mortar and pestle, new lumps were produced.

It was first suspected that adsorbed moisture was responsible for this condition. Accordingly, several grams were spread on the bottom of a beaker which was then placed in an oven at 105°C for two hours. When the material was later cooled, it was found that the color had deepened perceptibly and that the material had cinkered, i.e., the material was much harder and was more extensively agglomerated into large particles. Upon grinding the cinkered material with a mortar and pestle, the original color and small particle size was restored, but the original cohesive state was also restored. This condition has dominated the results obtained in our experiments with Day-Glo Orange powder.

Two experiments were conducted with Day-Glo Orange powder employing again the 21-liter flask at less than 1 mm Hg pressure, and allowing the rush of an air charge behind the powder to carry it into the evacuated flask and to disperse it there. In the first of these

(Experiment 12), a limited air charge was used to inject the powder into the flask from a chemical introduction chamber with outer end closed. This was followed by allowing the external air to rush in through another opening, tangent to the vessel wall, thereby producing a vortex-like motion of the powder within. The arrangement of the apparatus can be seen in figure 2. The structure of the glass apparatus through which the powder was introduced is shown in figure 3. The air in the outer end of the chamber propelled the powder through the 6-mm inside diameter hole of the stopcock plug. Because the lower end of this device had a choke effect, a second stratagem was employed.

In the second experiment (Experiment 21) the Day-Glo Orange powder was placed inside the flask on a 2.54-cm coarse-porosity glass fritted disc mounted as shown in figure 4. The apparatus is shown also in figure 5. The pressure inside the flask was then lowered to less than 1 mm Hg simultaneously on both sides of the fritted disc. After evacuation of the flask, the stopcocks to the flask sidearm and vacuum line were closed and air was allowed to rush as rapidly as possible into the flask only through the sample. A much larger angle of trajectories of sample particles into the flask was achieved in this case than was achieved in the first experiment with Day-Glo Orange powder.

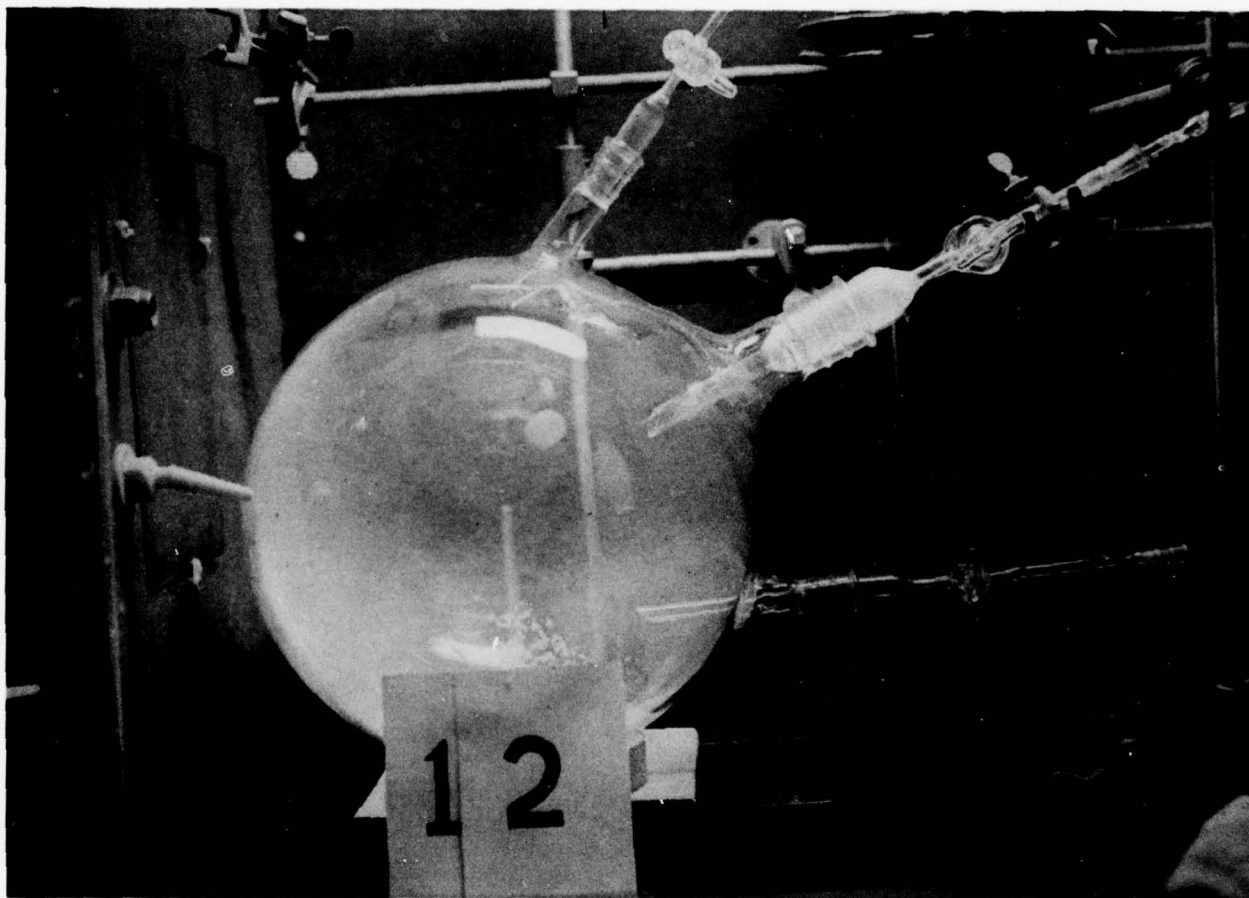


Figure 2. Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 before Introduction of Air into the Chamber



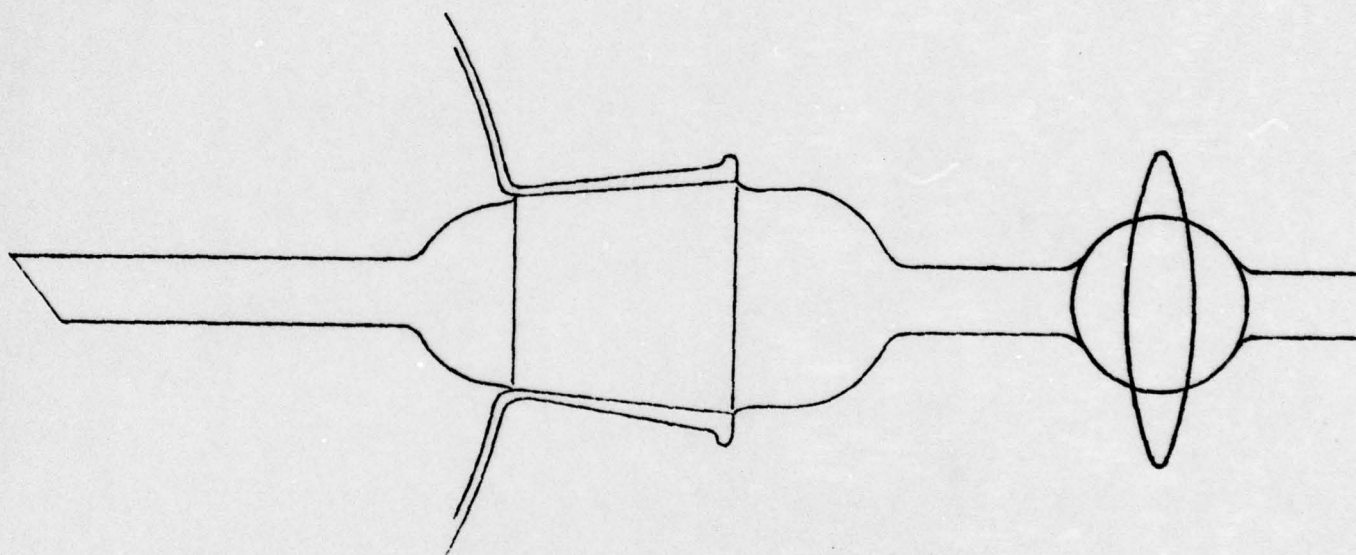


Figure 3. Glass Apparatus Arrangement Through Which Day-Glo Orange Powder was Introduced into Dispersion Chamber by the First Method.



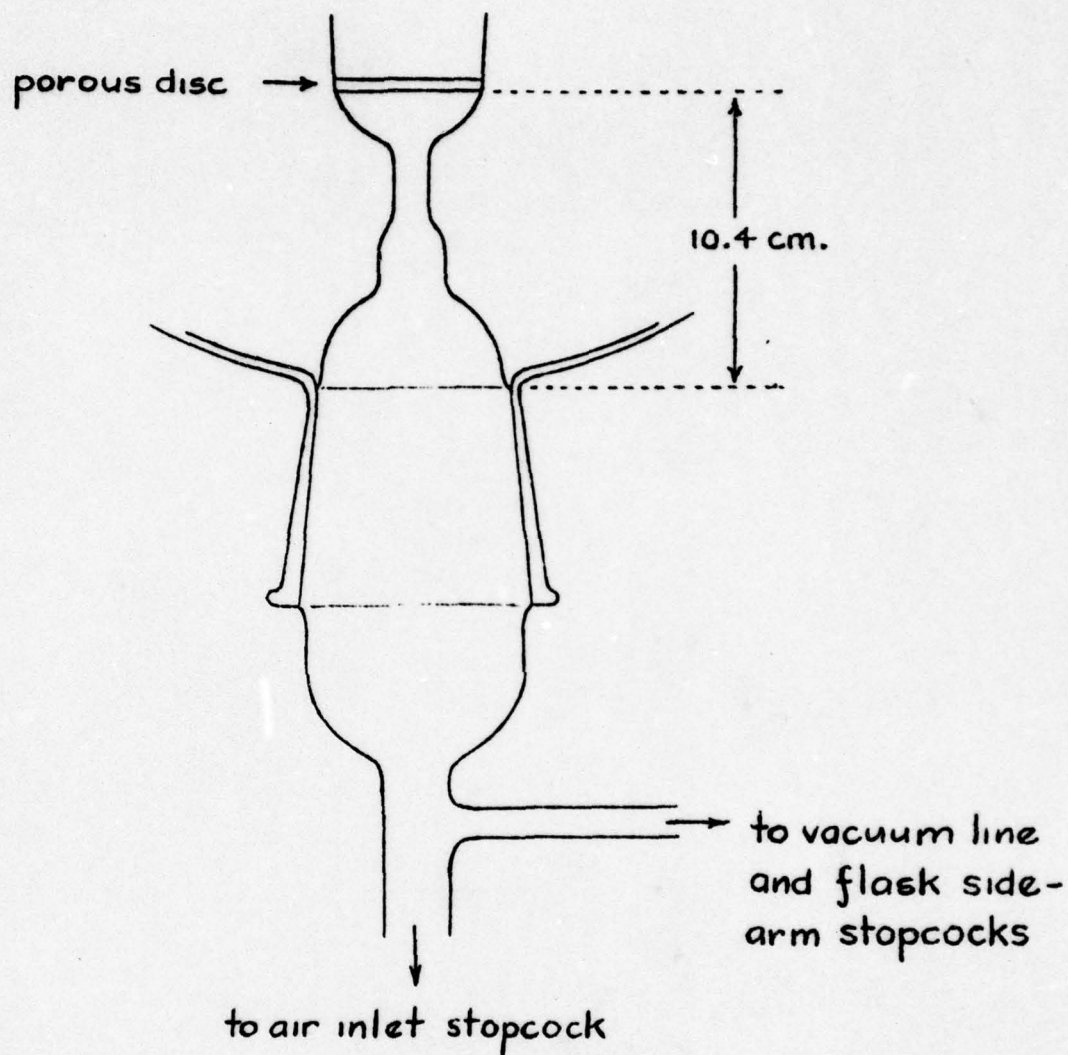


Figure 4. Glass Apparatus Arrangement With Which Day-Glo Orange Powder was Dispersed by the Second Method.



Figure 5. Apparatus used in Dispersion of Day-Glo Orange Powder by the Second Method in Experiment 21

### C. Procedure for Burning LTSB Mixtures

The apparatus used in burning LTSB Mixtures I and III is shown in figure 6. In these experiments the chemical introduction chamber with stopcock was attached to the flask through a standard taper joint to function only as a vacuum release valve. The Pyrex sidearm attached to the flask, as shown on the lower right of figure 6, was positioned horizontally and housed an LTSB mixture pellet at its end. The pellet was set in position before the pressure in the vessel was lowered. The pellet sidearm was 17.5 mm in inside diameter, and 22.0 mm in outside diameter.

When the pressure inside the apparatus had fallen below 1 mm Hg, a small tube furnace, having an inside temperature of 500°C at its center, was moved over the pellet sidearm so that the pellet was in the center of the furnace. The annulus between the sidearm and furnace cavity at its end was then quickly packed with a siliceous thermal insulation material. The other end of the furnace cavity was similarly closed at all times. About two minutes elapsed before the pellet temperature was sufficiently high for burning to start.

In the case of LTSB Mixture II, the temperature of the sample pellet could not be raised enough to cause ignition because of the Pyrex pellet sidearm (softening point about 550°C). Therefore, Mixture II was ignited in a porcelain crucible with heat applied from a hand torch, as shown in figure 7. The mouth of the crucible was somewhat smaller than that of the 45/50  $\frac{1}{2}$  mouth of the flask which was placed



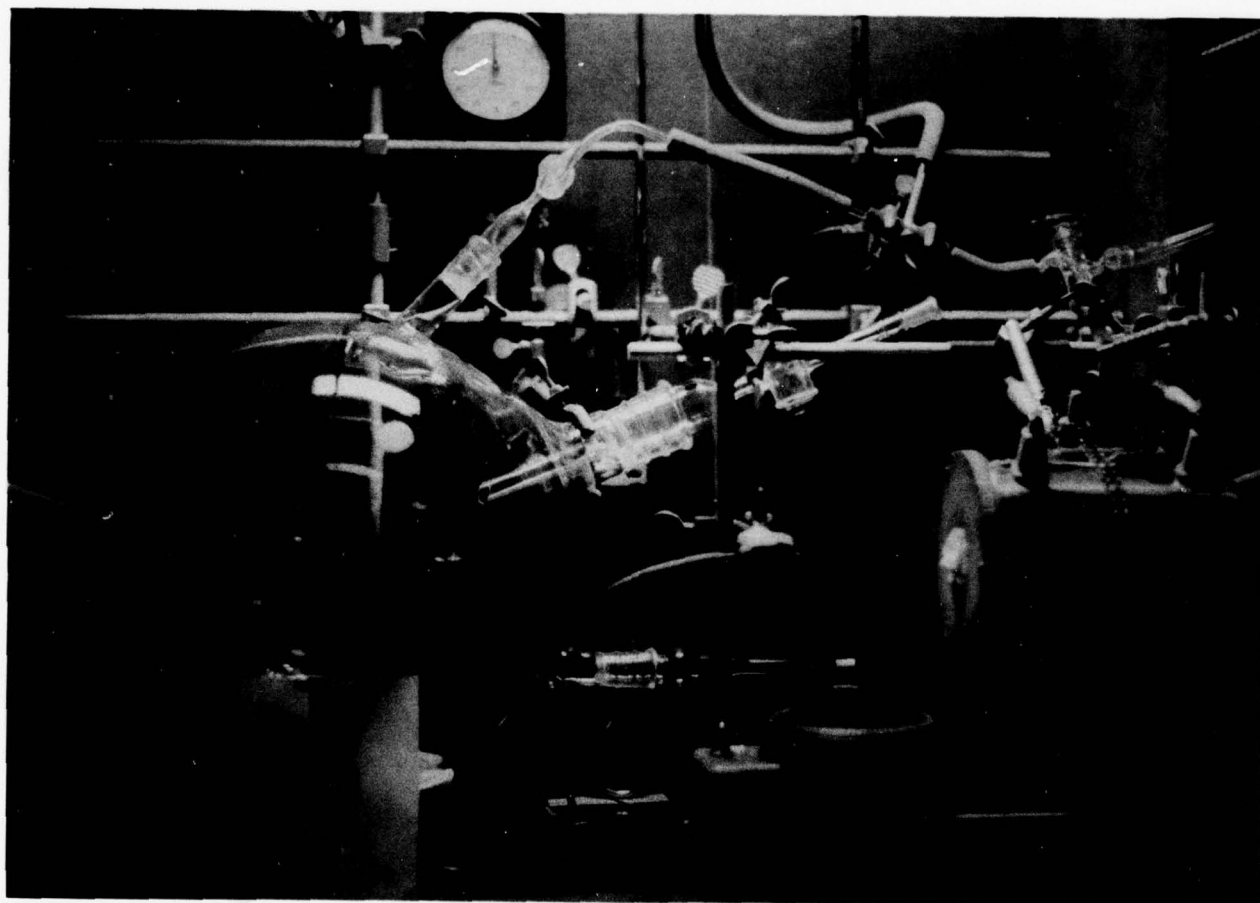


Figure 6. Apparatus for Generation of Smokes from LTSB Mixtures I and III in Experiments 13, 14, 15, 16, and 17

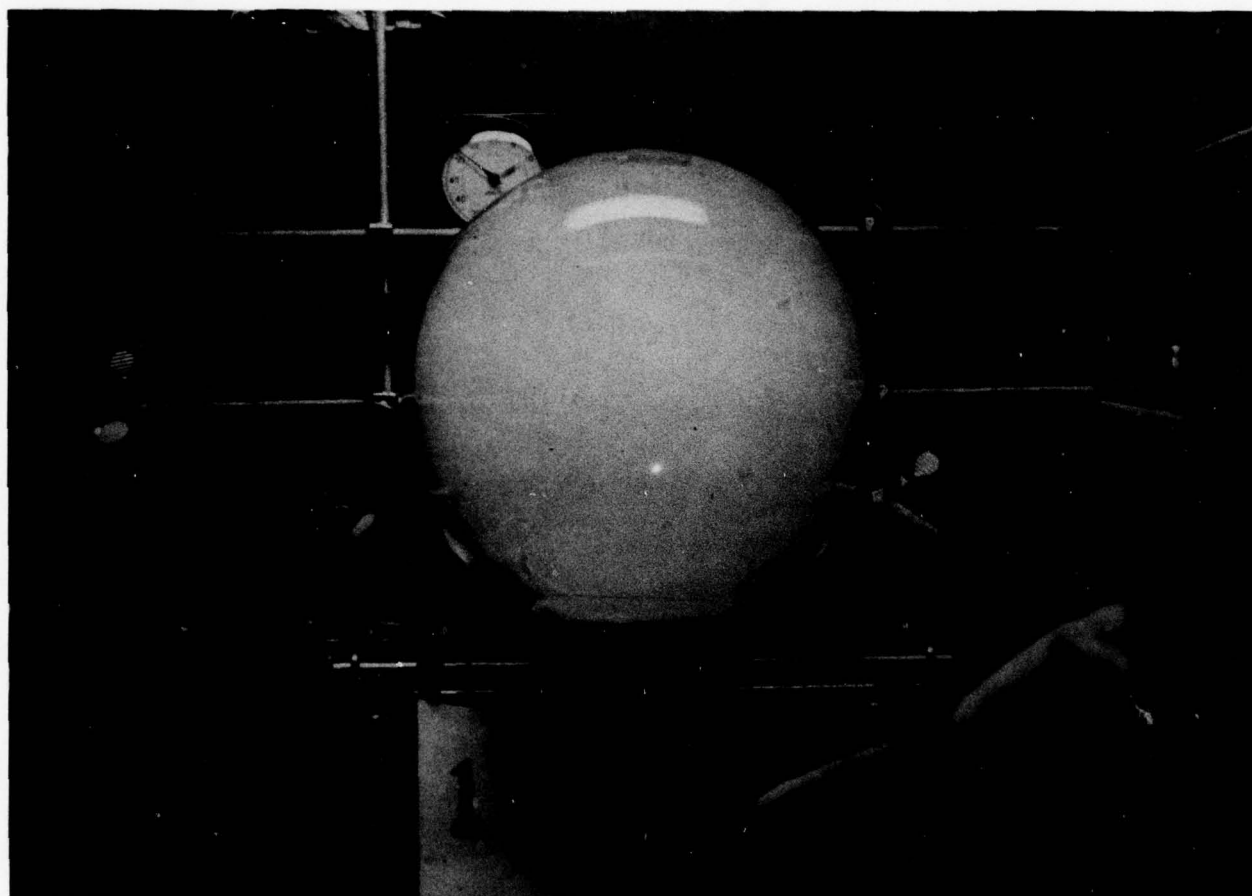


Figure 7. Smoke at Conclusion of Burning LTSC Mixture II in Experiment 19

over the crucible mouth. See figure 8. The other openings of the flask were suitably occupied by an open eductor tube in one case, and a glass stopper in the other case (left side of figures 7 and 8).

## 2. Chemicals and Chemical Mixtures

The following chemicals were used in the experiments described in this report.

- (1) Acetyl chloride, Baker Analyzed Reagent, Lot No. 41449, J. T. Baker Chemical Co., Phillipsburg, NJ.
- (2) Aluminum powder (USP), AX630, CB 112, Matheson-Coleman and Bell.
- (3) Ammonium hydroxide, 28% by weight, Analytical Reagent, 3256, Mallinckrodt, Inc., St. Louis, MO.
- (4) Confectionary sugar, Godchaux-Henderson Sugar Co., Reserve, LA.
- (5) Day-Glo Orange powder, unknown origin. Obtained from BEI Electronics, Inc., Little Rock, AR.
- (6) Hexachloroethane, 8028-352364, Matheson-Coleman and Bell, The Matheson Co., Norwood, OH.
- (7) Potassium chlorate, unknown origin.
- (8) Sodium bicarbonate, Lot No. 750971 Fisher certified reagent, Fisher Scientific Co., Fair Lawn, NJ.
- (9) Sulfur, flowers of, unknown origin.
- (10) Sulfuryl chloride, Practical, P-322, Eastman Organic Chemicals, Eastman Kodak Co., Rochester, NY.
- (11) 1,4,5,8-tetrachloroanthraquinone, FFB, G-3771, T-294, 78A0620, Bayer AG Leverkusen.
- (12) Titanium (IV) chloride, 99.9%, Lot No. 090778, Ventron, Alfa Division, Danvers, MA.
- (13) Uvinul 400, Lot No. 76927, GAF Corporation, New York, NY.
- (14) Zinc oxide, Fisher certified reagent, Lot No. 702053, Fisher Scientific Co., Fair Lawn, NJ.



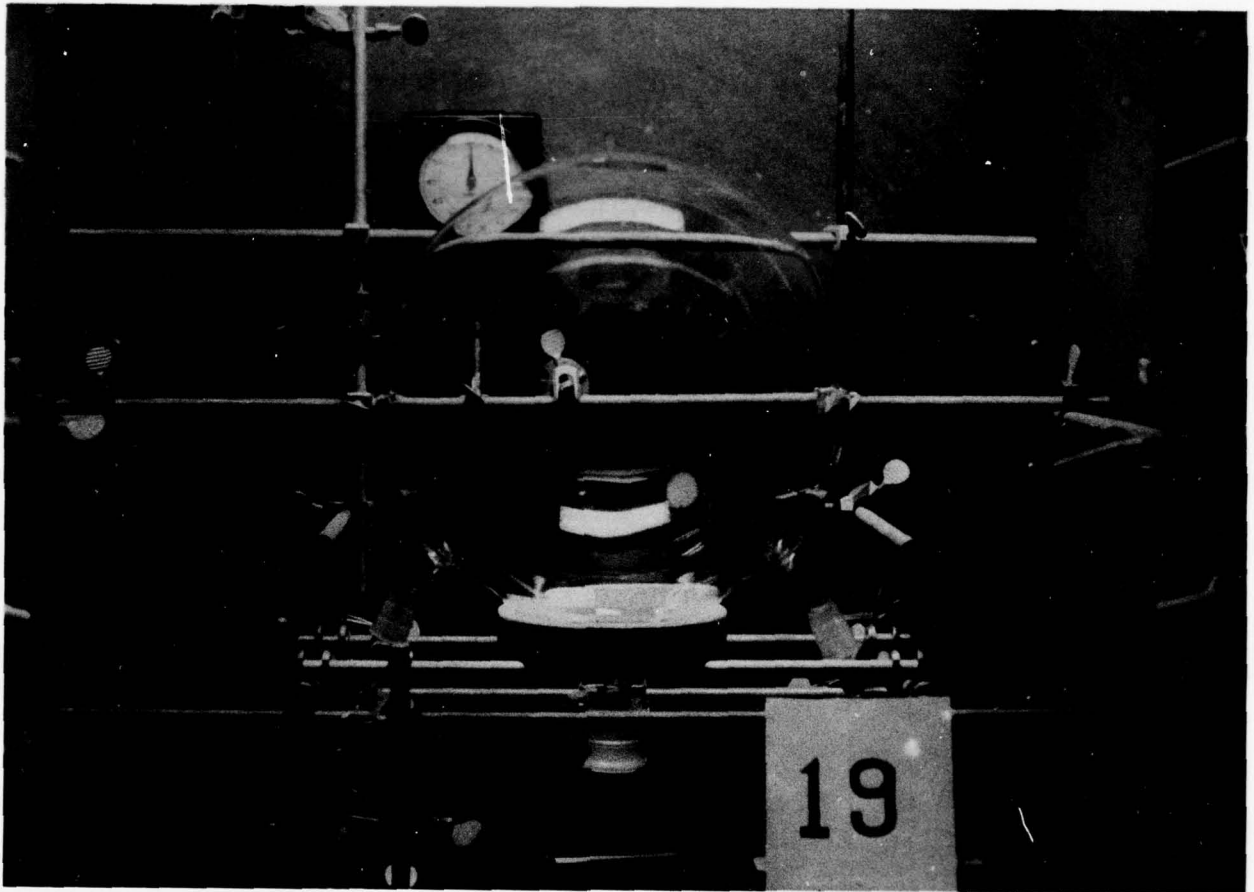


Figure 8. Apparatus for Burning LTSC Mixture II in Experiments 19 and 20

The LTSB Mixtures were composed as listed in Table I.

Table I. LTSB Mixtures Compositions

Mixture	Material	Weight Percent
I	sulfur	8.8
	sodium bicarbonate	24.5
	potassium chlorate	22.5
	Uvinul 400	44.2
II	zinc oxide	46.5
	hexachloroethane	44.5
	aluminum powder	9.0
III	potassium chlorate	35.0
	sugar	35.0
	1,4,5,8-tetrachloro-	
	anthraquinone	30.0

A 40-gram batch of each of these mixtures was prepared. Each batch was milled in a milling jar for five hours. A sample of each of Mixtures I and II was then pelletized in a stainless steel die using a pressure of 3000 pounds per square inch maintained for 60 seconds. In the case of Mixture III, the material was very soft and putty-like. Therefore, a cylindrical pellet was hand-shaped about the same size as those obtained for Mixtures I and II. Dimensions and weights of pellets are given in the following text.

### 3. Photographs and Other Data Forms

The number appearing within a photograph refers to an experiment, and gives the order in which the experiment was performed. In some cases several photographs were taken during a single experiment.

#### 4. Experimental Results and Discussion

##### A. Spontaneous Reaction between Sulfuryl Chloride and Aqueous Ammonia - Experiments 1, 2, 3, and 4

In each of these experiments, the amount of each of the reacting chemicals used was based upon the equation:



The chemicals, in order of appearance in this equation, are: sulfuryl chloride, ammonia, water, ammonium chloride, and ammonium sulfate. This is an idealization of what takes place. Side reactions may occur, or the reaction described by the equation may occur in a stepwise manner. The principal overall reaction is, nevertheless, the one represented.

The aqueous ammonia was 28-percent ammonia by weight with a density of 0.90 g/ml. The density of sulfuryl chloride is about 1.667 g/ml. These values were used to calculate the desired weights of reactants. In most of these experiments, the intent was to use a total volume of 4 ml of chemicals. Using this volume, and the stoichiometry of the chemical equation given above as constraints, the weight of 28-percent aqueous ammonia required is  $2.77\text{g}(Q_1)$ , and that of sulfuryl chloride is  $1.53\text{g}(Q_2)$ . Their respective volumes are 3.08 ml and 0.92 ml. For this idealized reaction,  $Q_1/Q_2 = 1.81$ . Hereafter  $Q_1/Q_2$  is designated R.

Table II gives the conditions under which 1, 2, 3, and 4 were conducted. In all of these experiments the prereaction pressure in the vessel was 1 mm Hg.



Table II. Conditions Employed in Experiments  
1, 2, 3, and 4. Reacting Sulfuryl Chloride and  
Aqueous Ammonia

Figure	Expt. No.	$Q_1$ , Wt. $\text{NH}_4\text{OH}$ (g)	$Q_2$ , Wt. $\text{SO}_2\text{Cl}_2$ (g)	$Q_1/Q_2$ (R)	Total Vol ml	Ambient Temp $^{\circ}\text{C}$
9	1	4.32	5.08	0.85	7.85	26
10	2	2.74	1.65	1.66	4.03	24
11	3	2.81	1.49	1.89	4.01	25
12	4	2.76	1.57	1.76	4.01	24

In this set of experiments the R value has a wide range. The very low value of 0.85 in Experiment 1 was used to see if the unaided eye could detect a more rapid development of smoke than was observed in preliminary experiments where R was closer to 1.81, and total volume of chemicals was much less than 7.85 ml. Such a difference was not detectable. Figure 9 cannot be compared with figures 10, 11, and 12, as it is the result of a photographic exposure that is different from that of the latter three photographs.

In comparing figures 10, 11, and 12, the quality of smoke appears to diminish in the order figure 10 > figure 12 > figure 11. This is the same order as increasing value of R. This seems to indicate that at R values in excess of 1.81 the quality of smoke from this reaction mixture, under these experimental conditions, is poorer than at R values below 1.81. However, this can only be proved by more elaborate experiments which more quantitatively characterize the smoke produced from the standpoint of rate of change of optical density and uniformity within the reaction vessel.

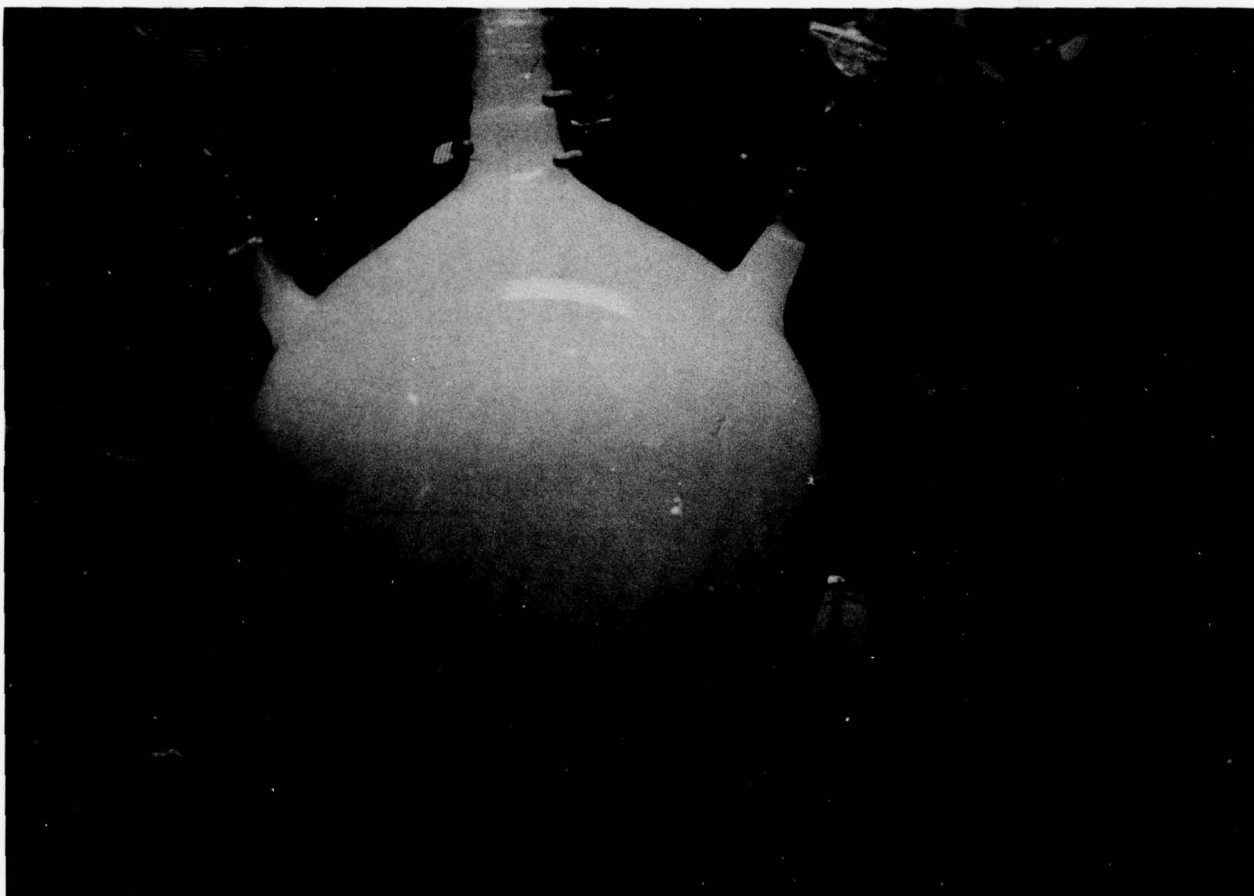


Figure 9. Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 1

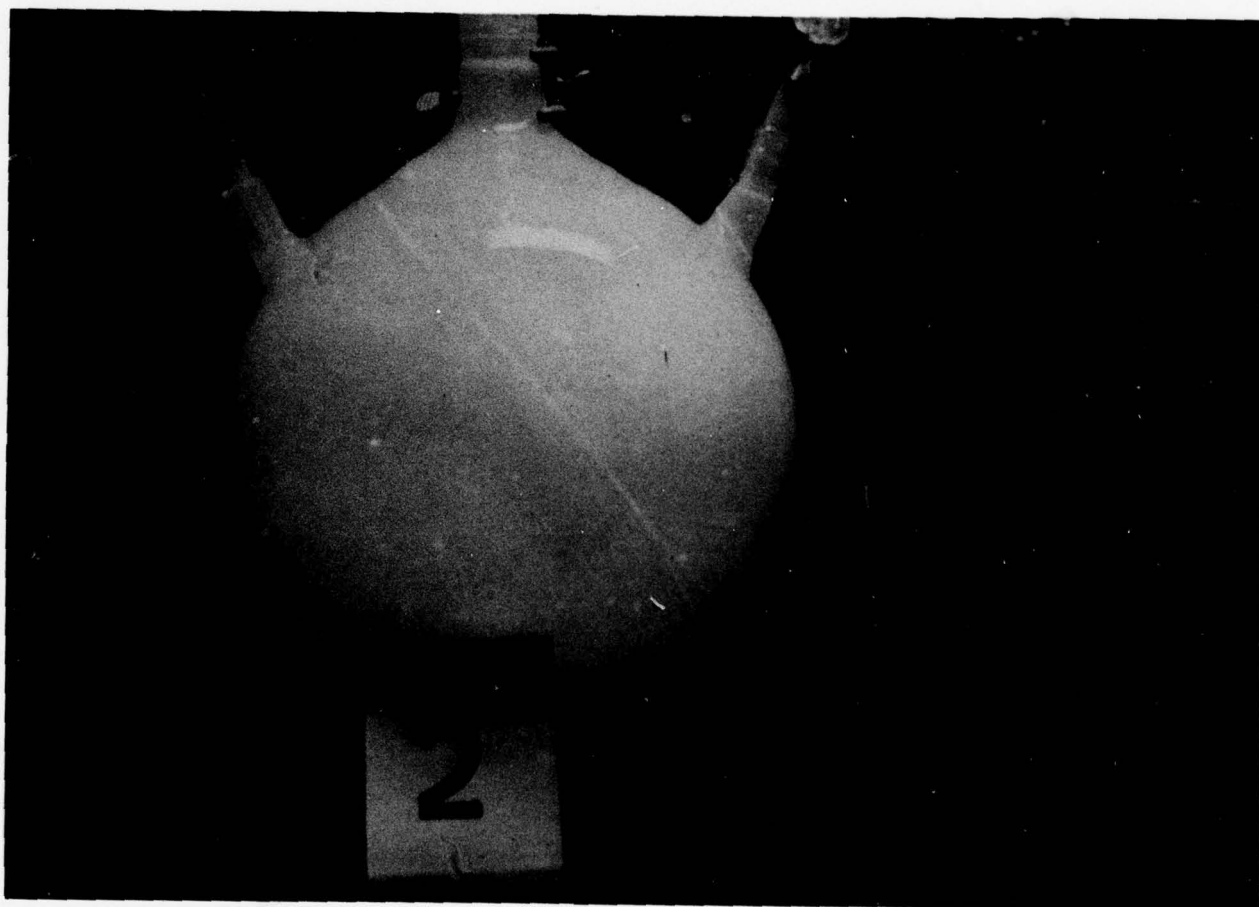


Figure 10. Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 2



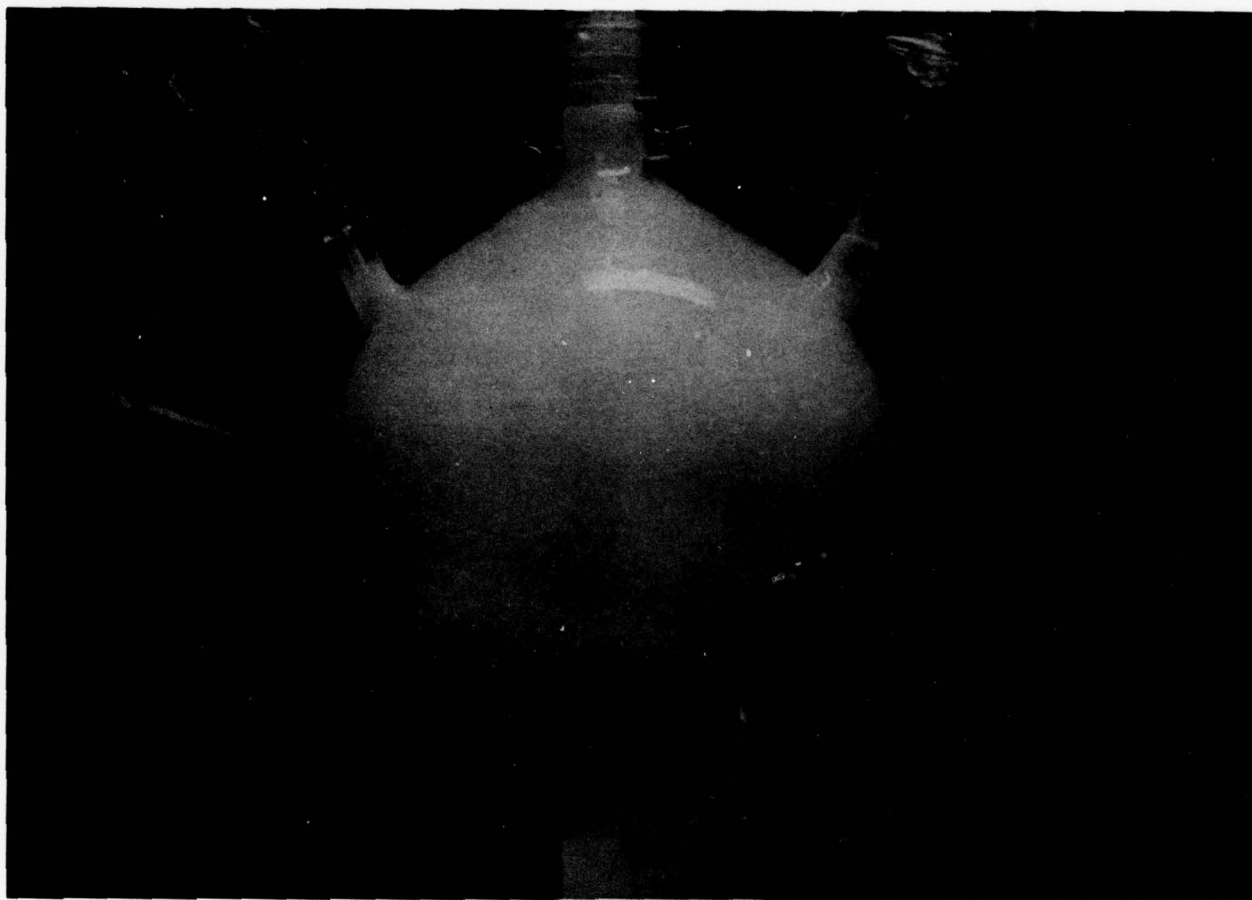


Figure 11. Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 3

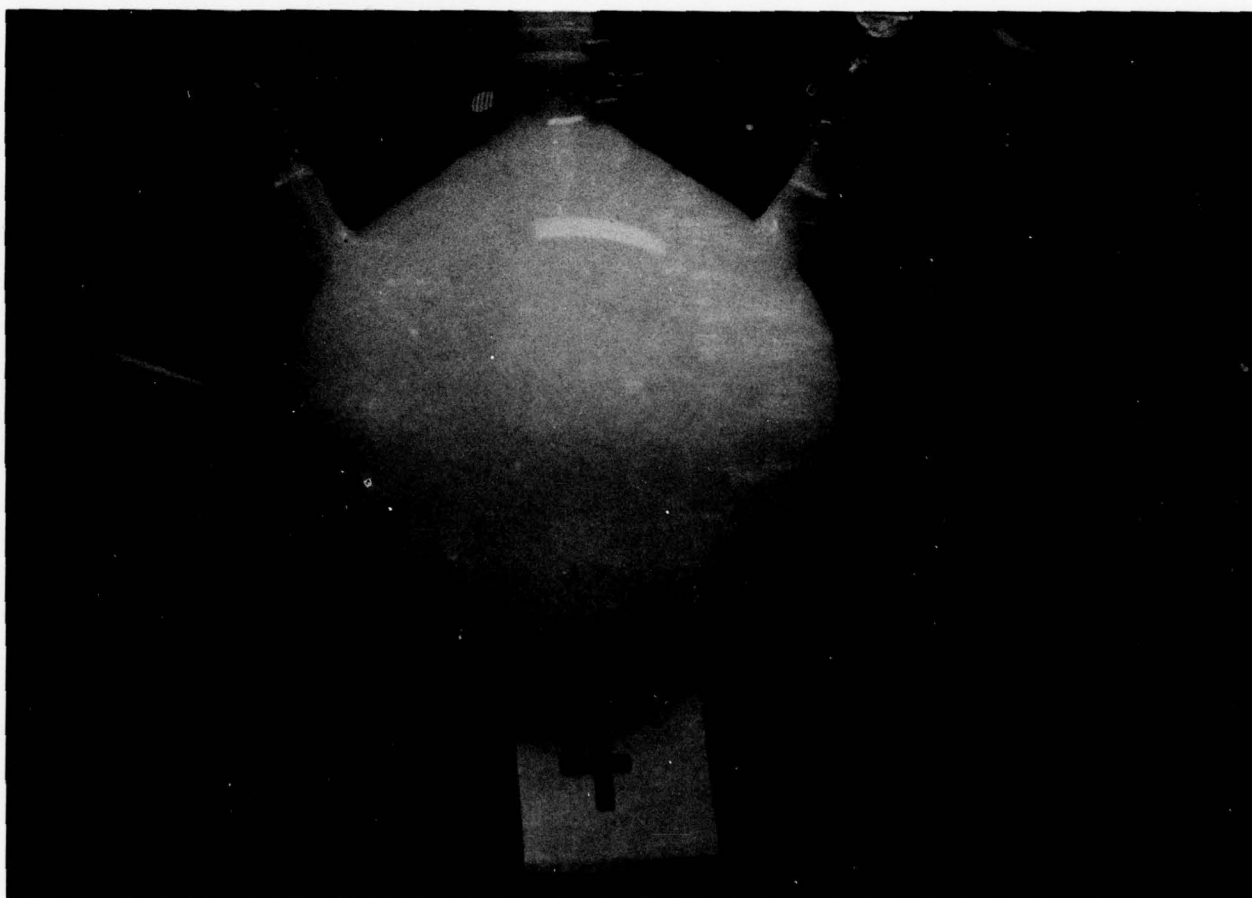
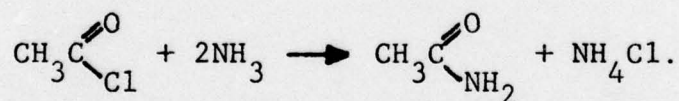


Figure 12. Smoke from Reaction of Sulfuryl Chloride and Ammonium Hydroxide in Experiment 4

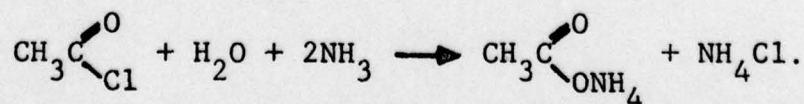
It should be pointed out that the first-hand observations of the development of the smokes photographed has shown that photographs can be misleading about the quality of smoke produced. For example, the reaction of aqueous ammonia ( $\text{NH}_4\text{OH}$ ) and acetyl chloride ( $\text{CH}_3\text{COCl}$ ) was studied. The results of this are shown in figures 13, 14, and 15. Comparison of these photographs with figures 10, 11, and 12, for the  $\text{NH}_4\text{OH}$ ,  $\text{SO}_2\text{Cl}_2$  reaction mixture shows a much better quality smoke produced from  $\text{SO}_2\text{Cl}_2$  than from  $\text{CH}_3\text{COCl}$ . However, the disparity between the two as observed with the unaided eye was greater than is apparent by comparing the photographs. Before the  $\text{CH}_3\text{COCl}$  experiments, it was anticipated that the quality of smoke would be poorer than obtained with  $\text{SO}_2\text{Cl}_2$ .

B. Spontaneous Reaction between Acetyl Chloride and Aqueous Ammonia - Experiments 5, 6, and 7

In this case the assumed overall reaction is represented by:



The chemicals, in order of appearance in the equation are: acetyl chloride, ammonia, acetamide, and ammonium chloride. Side reaction is also a possibility here, the principal one being:



The density of  $\text{CH}_3\text{COCl}$  used in the calculations was 1.105 g/ml. The weight of 28-percent aqueous ammonia required was 2.37g ( $Q_1$ ), and



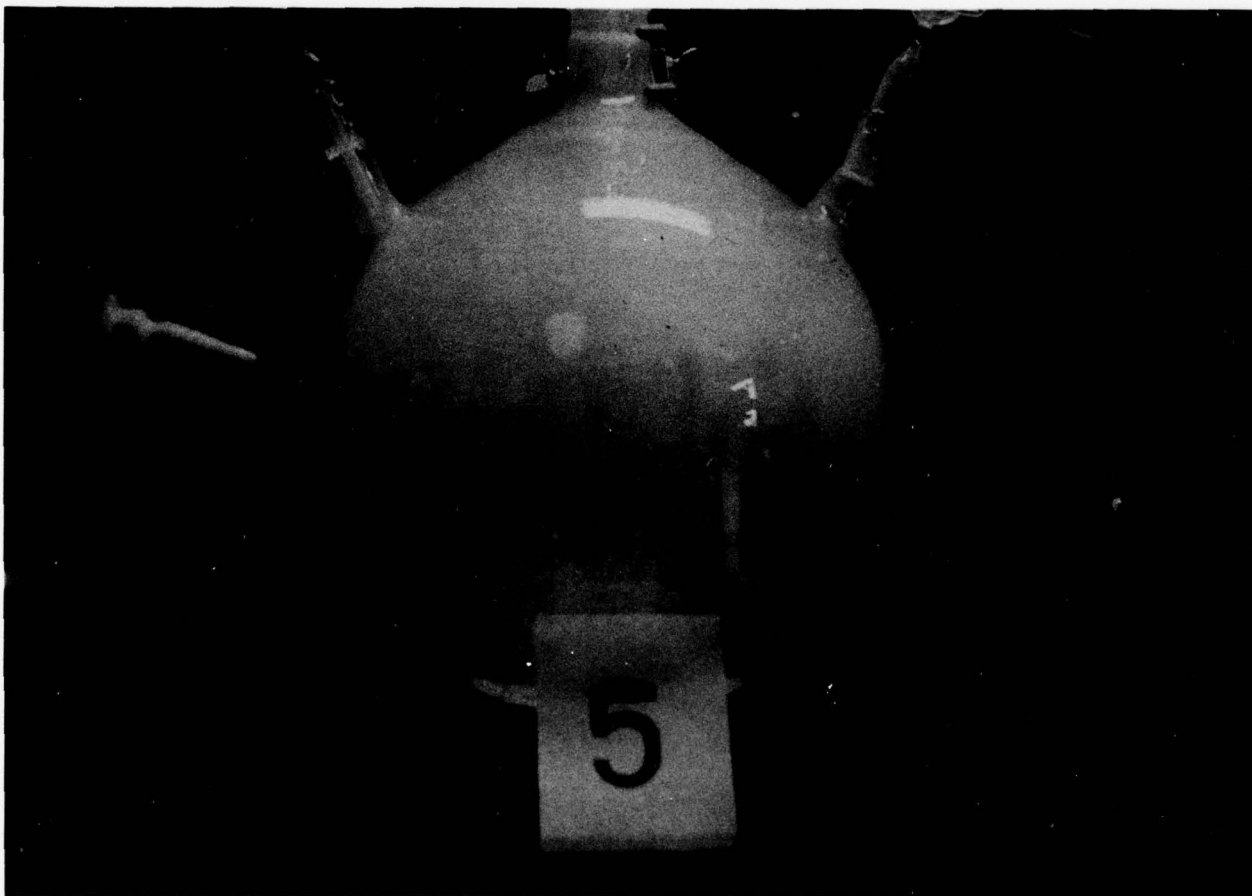


Figure 13. Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 5

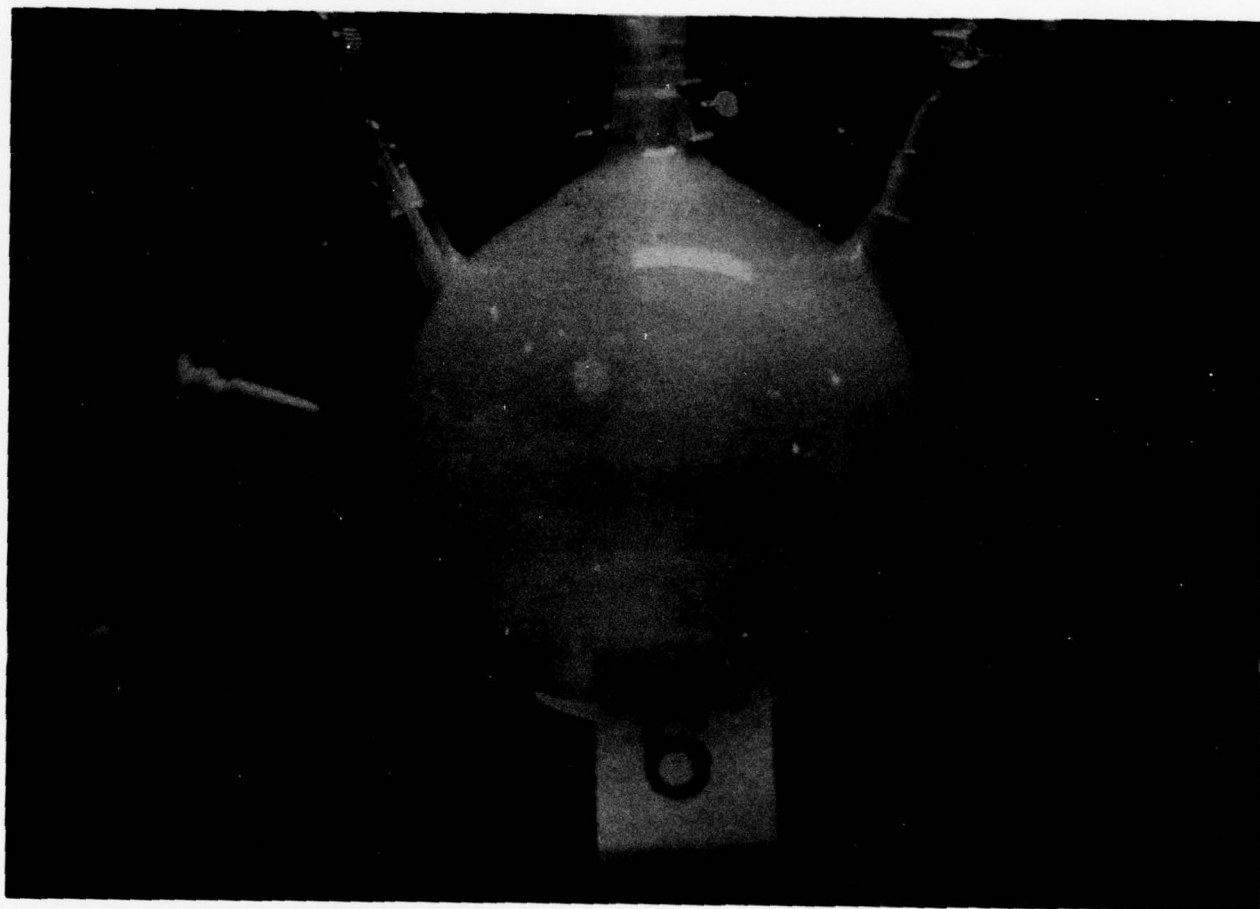


Figure 14. Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 6

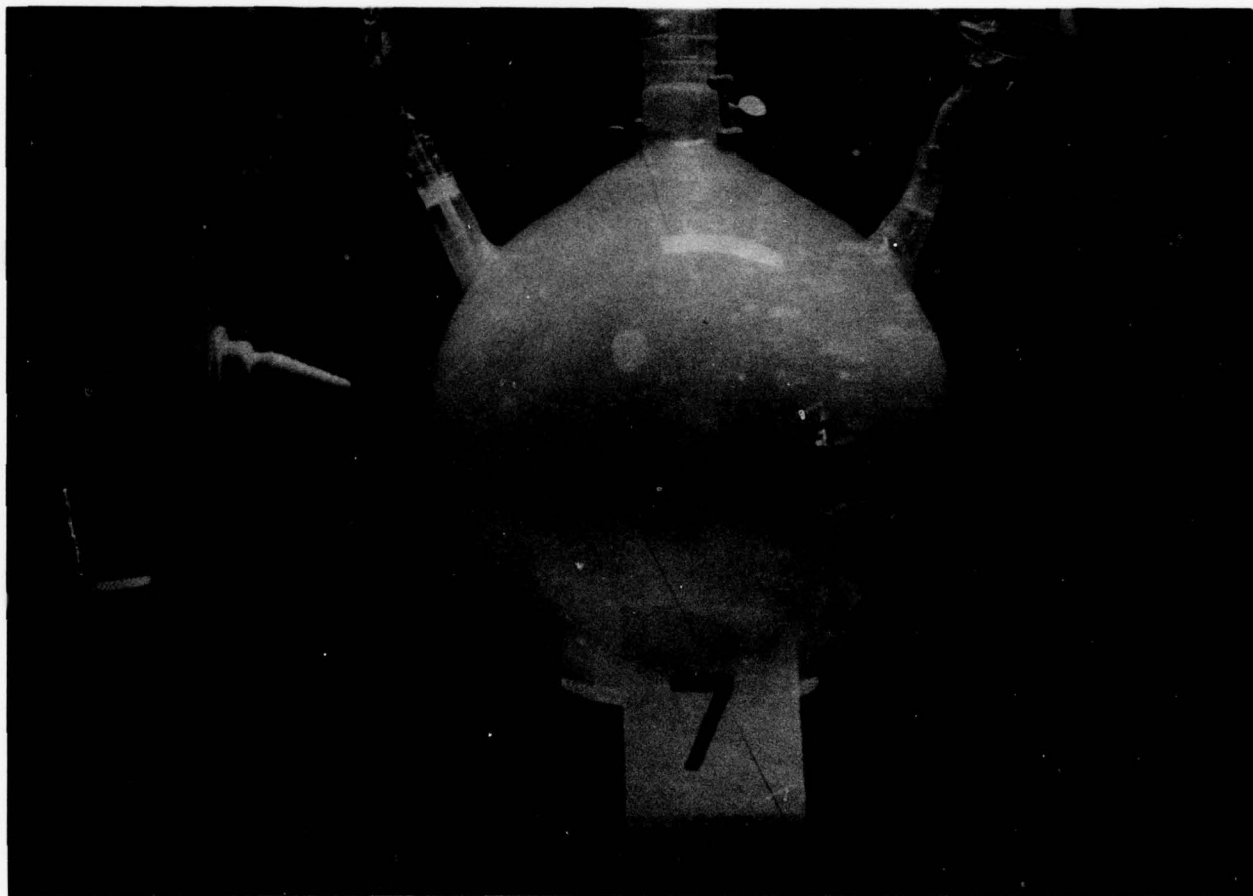


Figure 15. Smoke from Reaction of Acetyl Chloride and Ammonium Hydroxide in Experiment 7



that of  $\text{CH}_3\text{COCl}$  was 1.51g ( $Q_2$ ). Their respective volumes were 2.63 ml and 1.37 ml. For this idealized reaction  $Q_1/Q_2 = R = 1.57$ .

Table III gives the conditions under which experiments 5, 6, and 7 were conducted. The evacuated flask was again employed.

Table III. Conditions Employed in Experiments 5, 6, and 7.  
Reacting Acetyl Chloride and Aqueous Ammonia.

Figure	Expt. No.	$Q_1$ , Wt $\text{NH}_4\text{OH}$ (g)	$Q_2$ , Wt $\text{CH}_3\text{COCl}$ (g)	R	Total Vol ml	Ambient Temp $^{\circ}\text{C}$
13	5	2.42	1.56	1.55	4.10	24
14	6	2.42	1.65	1.47	4.18	24
15	7	2.50	1.63	1.53	4.26	25

Whereas the total volume of liquid chemicals used in each of the A and B sets of experiments was about 4.00 ml, the number of moles of reactants was different by a substantial factor. In experiment 2 where 1.65 g of  $\text{SO}_2\text{Cl}_2$  was used, 0.0122 moles of  $\text{SO}_2\text{Cl}_2$  was used. In experiment 7 where 1.65 g of  $\text{CH}_3\text{COCl}$  was used, the corresponding number of moles was 0.0210.

C. Spontaneous Reaction between Titanium Tetrachloride and Water - Experiments 8, 9, 10 and 11

The assumed overall reaction in this case is represented by:



Once again, side reactions and stepwise reaction are possible. This is more likely than in the previous cases. It is quite likely that some of the product would be hydrous titanium oxide with the formula  $\text{TiO}_2 \cdot x\text{H}_2\text{O}$  where  $x$  would have a range of values. The substances, in the order of appearance in the equation above, are: titanium tetrachloride (titanium (IV) chloride), water, titanium dioxide, and hydrogen chloride.

The density of  $\text{TiCl}_4$  used in the calculations based upon the equation above was 1.726 g/ml. The weight of water required was 0.98 g ( $Q_1$ ), and that of  $\text{TiCl}_4$  was 5.21 g ( $Q_2$ ). Their volumes were 0.98 ml and 3.02 ml respectively. Here  $Q_1/Q_2 = R = 0.188$ .

Table IV gives the conditions under which experiments 8, 9, 10, and 11 were conducted. The results are shown in figures 16, 17, 18, and 19.

Table IV. Conditions Employed in  
Experiments 8, 9, 10, and 11  
Reacting Titanium Tetrachloride and Water

Figure	Expt. No.	$Q_1$ , Wt $\text{H}_2\text{O}$ (g)	$Q_2$ , Wt $\text{TiCl}_4$ (g)	R	Total Vol ml	Ambient Temp $^{\circ}\text{C}$
16	8	1.00	5.55	0.180	4.22	24
17	9	1.00	5.55	0.180	4.22	24
18	10	0.33	1.83	0.180	1.41	22
19	11	0.33	1.92	0.172	1.44	21

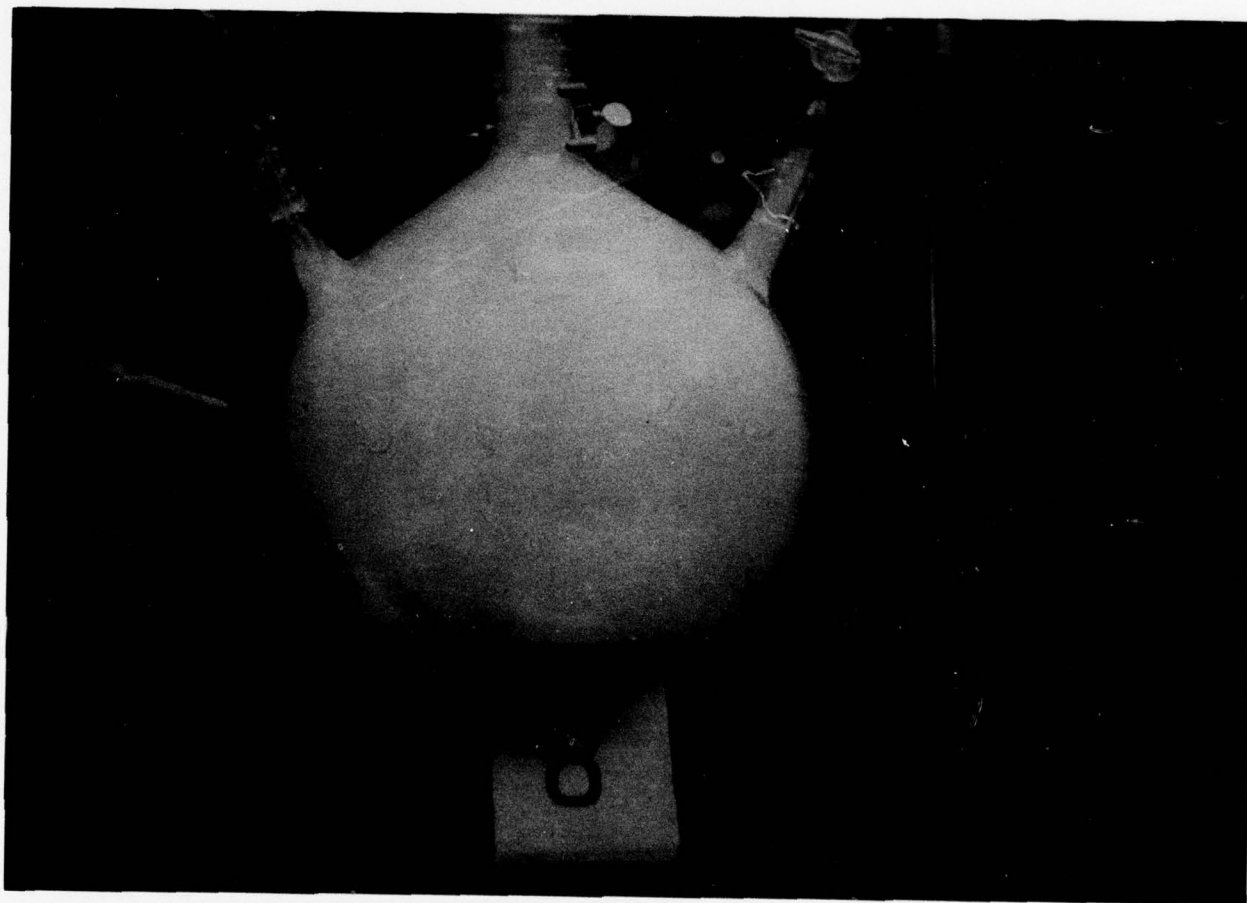


Figure 16. Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 8



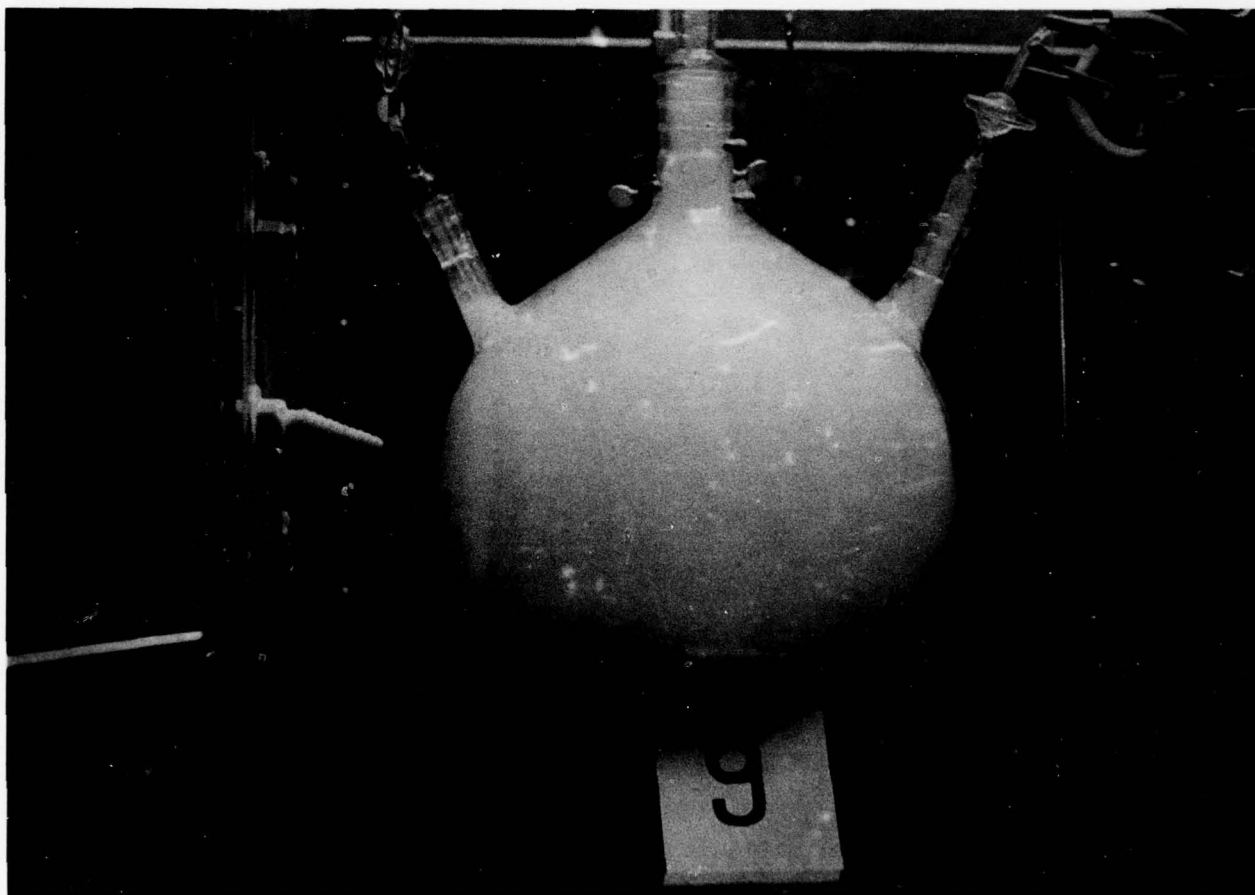


Figure 17. Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 9

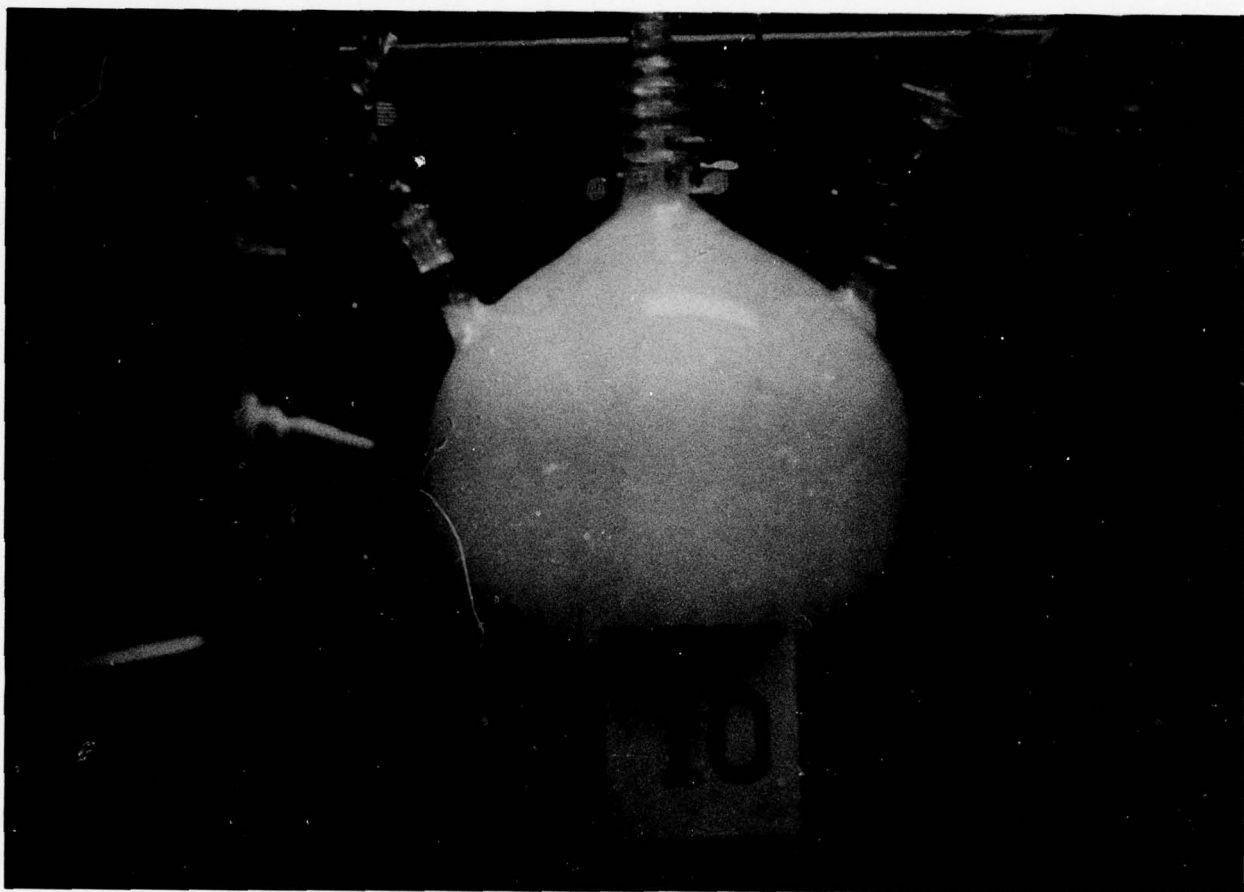


Figure 18. Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 10

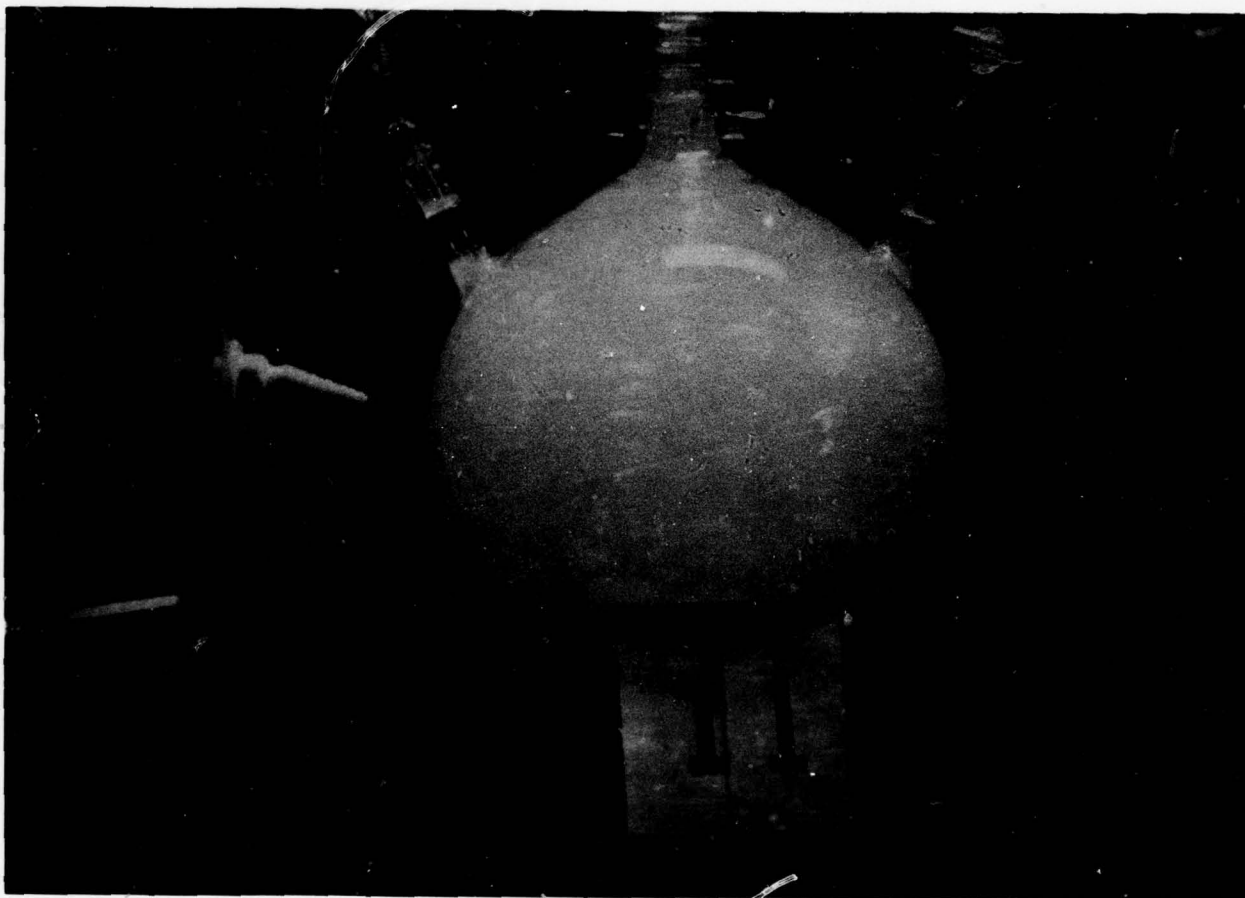


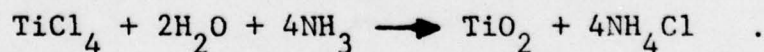
Figure 19. Smoke from Reaction of Titanium Tetrachloride and Water in Experiment 11



In experiments 8 and 9, the total volume of liquid chemicals used was about three times that used in experiments 10 and 11. The reason for this difference was that in 8 and 9 much of the  $\text{TiCl}_4$ , which was introduced into the flask first, did not vaporize before the water was introduced. However, the quality of smoke in these was the best obtained to that point. In experiments 10 and 11, very little of the  $\text{TiCl}_4$  remained liquid when the water was added. The quality of smoke in these two cases was as good as in experiments 8 and 9. The quantity was less in experiments 10 and 11 than in 8 and 9, as was expected.

In comparing the results of the series C experiments with those from experiment 2 (figure 10) from series A, it appears from the photographs that the results with sulfuryl chloride were better than those with  $\text{TiCl}_4$ . This is misleading. In a much larger space in which all  $\text{TiCl}_4$  could be vaporized, the quality and quantity of smoke would probably be much better than that observed first-hand. Nevertheless, under the conditions used in the experiments,  $\text{TiCl}_4$  smoke was superior to  $\text{SO}_2\text{Cl}_2$  smoke.

A spontaneous reaction between titanium tetrachloride and aqueous ammonia was conducted. This experiment (experiment 22) was similar to the other spontaneous reactions. The assumed reaction was:



Calculated requirements for a total liquid chemicals volume of 4.00 ml are: 2.57 g ( $Q_1$ ) of 28-percent aqueous ammonia and 1.98 g ( $Q_2$ ) of  $\text{TiCl}_4$ . Thus  $Q_1/Q_2$  is 1.30. The volumes are 2.85 ml and 1.15 ml,

respectively. The calculation is based upon required  $\text{NH}_3$  and  $\text{TiCl}_4$ . The result is that  $\text{H}_2\text{O}$  would be present in excess of that actually needed.

In the experiment as conducted, the weight of aqueous ammonia was 2.67 g ( $Q_1$ ), and that of  $\text{TiCl}_4$  was 2.30 g ( $Q_2$ ). The corresponding volumes were 2.97 ml and 1.33 ml, respectively. Total liquid chemicals volume used was 4.30 ml. The value of  $Q_1/Q_2$  was 1.16. Ambient temperature was  $24^\circ\text{C}$ .

The result of the experiment is shown in figure 20. As expected, there was excellent smoke generation without a residual concentration of  $\text{HCl}$ .

D. Dispersion of Day-Glo Orange Powder With Air - Experiments 12 and 21

The apparatus and method of dispersing Day-Glo Orange powder into a cloud or smoke confined to a 21-liter flask has already been described. Furthermore, the cohesion of microcrystals of this material into large particles and lumps has already been discussed, and it has been indicated that this has largely dictated the results obtained.

Dispersion was attempted by two methods. The first method was employed in experiment 12. The results are shown in figures 2, 21, and 22.

Figure 2 shows the poor dispersion obtained immediately after opening the stopcock between the chemical introduction chamber and the flask. Much of the orange powder was stuck to the flask wall opposite the tube protruding into the flask from the introduction chamber. Another

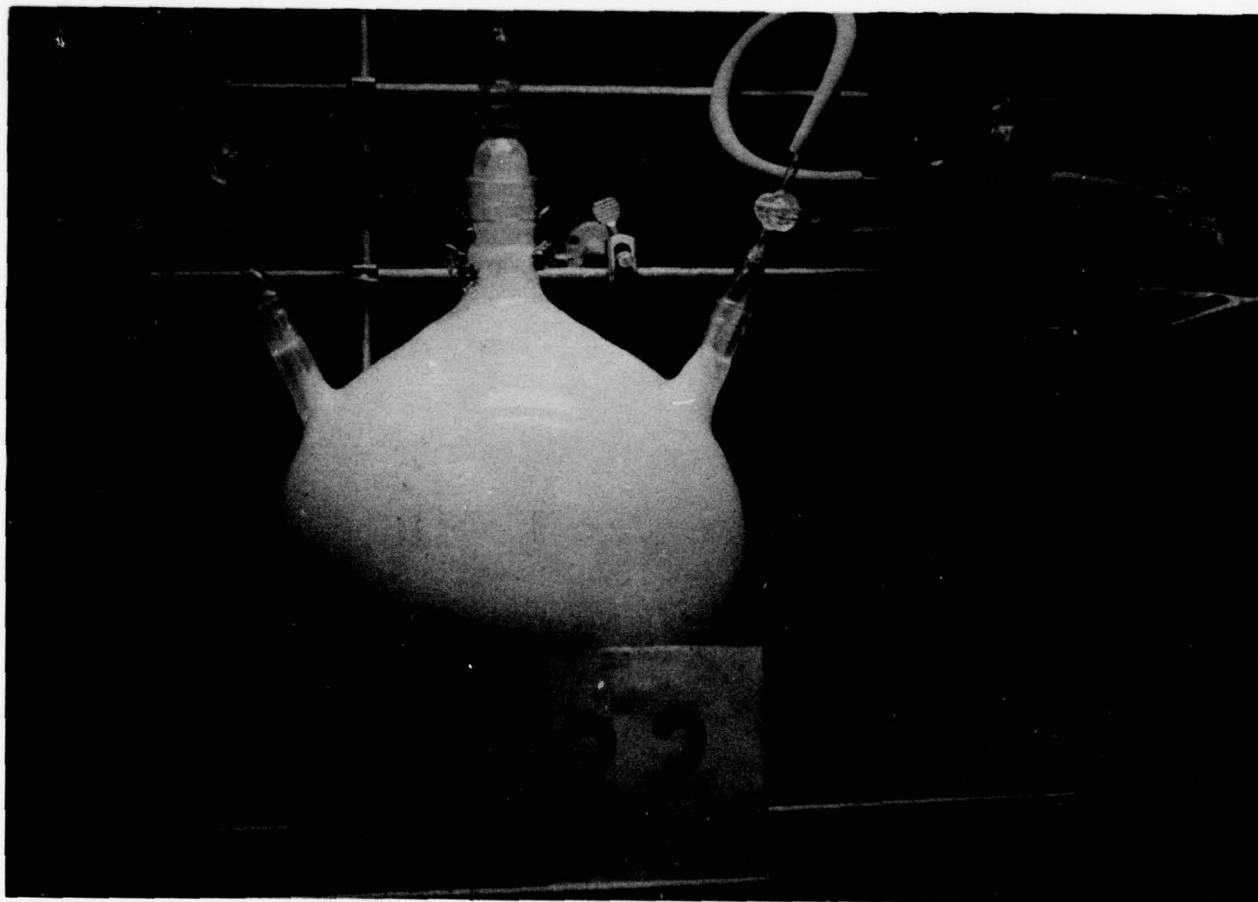


Figure 20. Smoke from Reaction of Titanium Tetrachloride and Aqueous Ammonia in Experiment 22



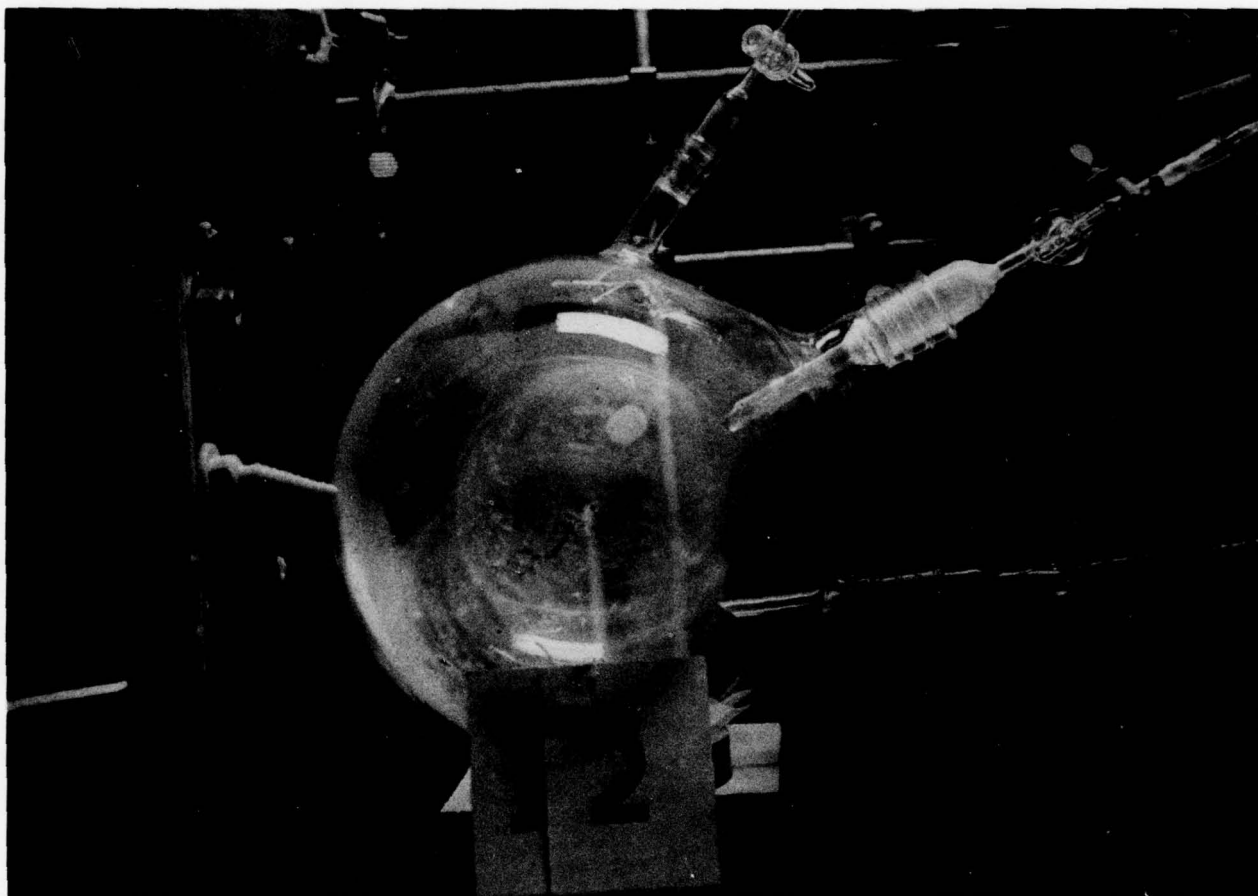


Figure 21. Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 while Introducing Air into the Chamber after Day-Glo Orange Introduction

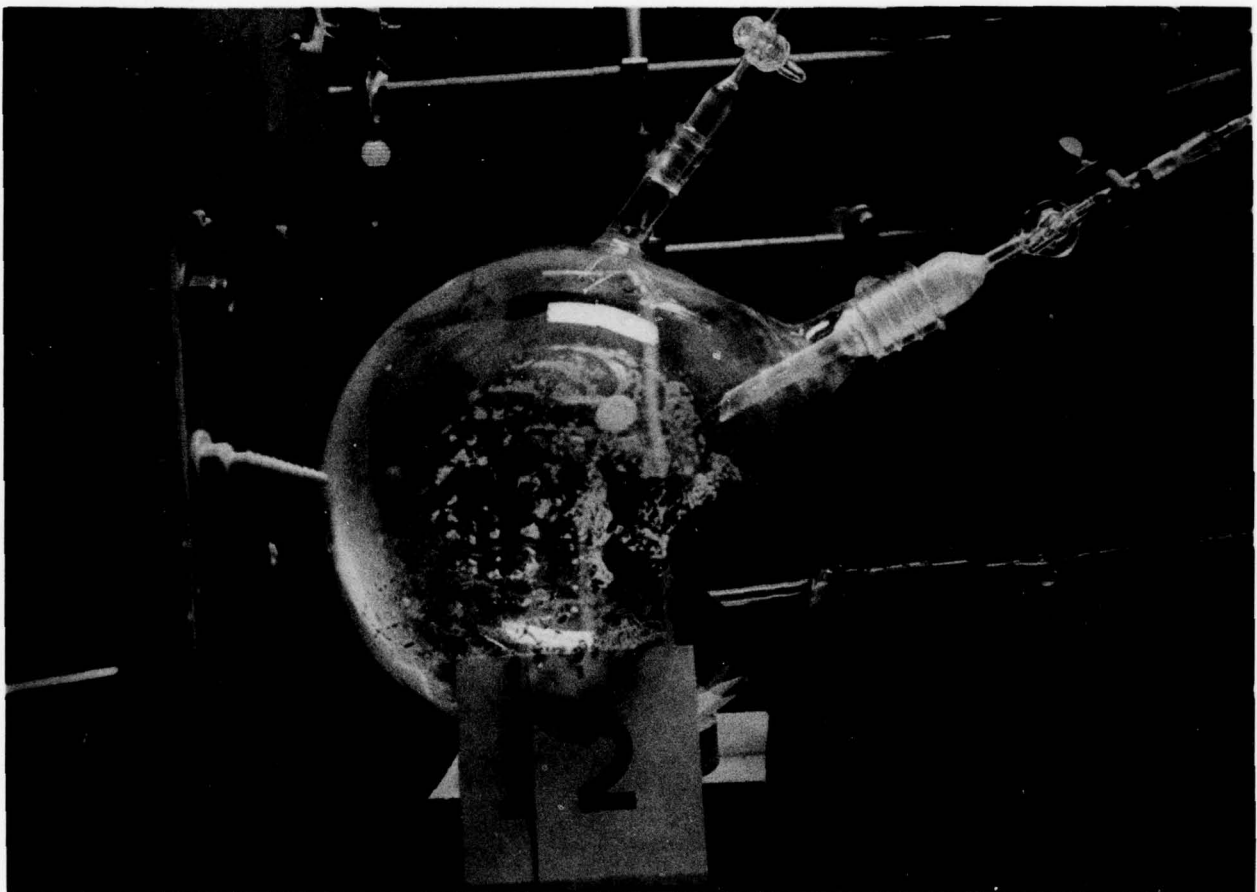


Figure 22. Dispersion of Day-Glo Orange Powder by the First Method in Experiment 12 after Bringing Pressure inside the Chamber up to One Atmosphere

large part lay in large lumps on the bottom wall of the flask, and only a small part of the powder, although more finely dispersed, nevertheless adhered to the flask walls.

Immediately after figure 2 was taken, the lower, horizontal, empty chemical introduction chamber stopcock was completely opened. The beveled lower end of this chamber protruded into the flask with the bevel being so positioned as to direct the inward rushing air more or less downward but also largely tangential to the lower flask wall. This is shown in figures 2, 21, and 22. The result was to sweep some of the powder on the flask walls into a swirling mass inside of the flask. This mass continued to swirl as long as the inside pressure was less than that outside of the flask. This is shown in figure 21. As soon as the air rush ceased, that powder that had not become attached to the side flask walls in the pressure equalization period fell to the bottom. Figure 22 shows clearly that a great portion of the powder was moved from the bottom of the flask to the side walls where it remained because of cohesion among powder particles and adhesion between powder and glass.

All of this illustrates the resistance of this material as received to dispersion into a cloud or smoke by means of an expanding gas mass. It may be possible to use the rush of a gas to disperse Day-Glo Orange powder, but the energy of the moving gas must be large enough to overcome the adhesive forces holding Day-Glo Orange microcrystals together. Even if this can be done, the microcrystal size may preclude the formation of a stable (persisting) dispersion because of rapid settling.



In the second method employed to disperse Day-Glo Orange powder in the flask, air moving between two spaces having a pressure differential of  $14.7 \text{ lb/in}^2$  was again employed. In this second case the air moved through the sample in such manner as to allow a much greater range of angle of trajectories of powder particles as they moved into the flask proper. The result was to more uniformly disperse the powder in the flask, but once again cohesive forces caused most of the material to move as lumps. The particles adhered to the flask walls, or quickly fell to the bottom of the apparatus. The result is shown in figure 23. This photograph is misleading in that the powder inside of the flask did not consist of suspended powder but rather of particles adhering to the walls. Furthermore, the distribution of powder on the walls was far less uniform than it appears to have been.

In Experiment 12, 1.30 g of powder, having a volume of 4 ml as measured with a 10 ml graduated cylinder, was used. The compaction of powder was that obtained by allowing the powder to fall no more than a distance of 15.3 cm.

The powder sample used in experiment 21 was also 1.30 g and was loosely spread on the supporting fritted glass disc.

The Day-Glo Orange powder is the least satisfactory of all of the materials used.

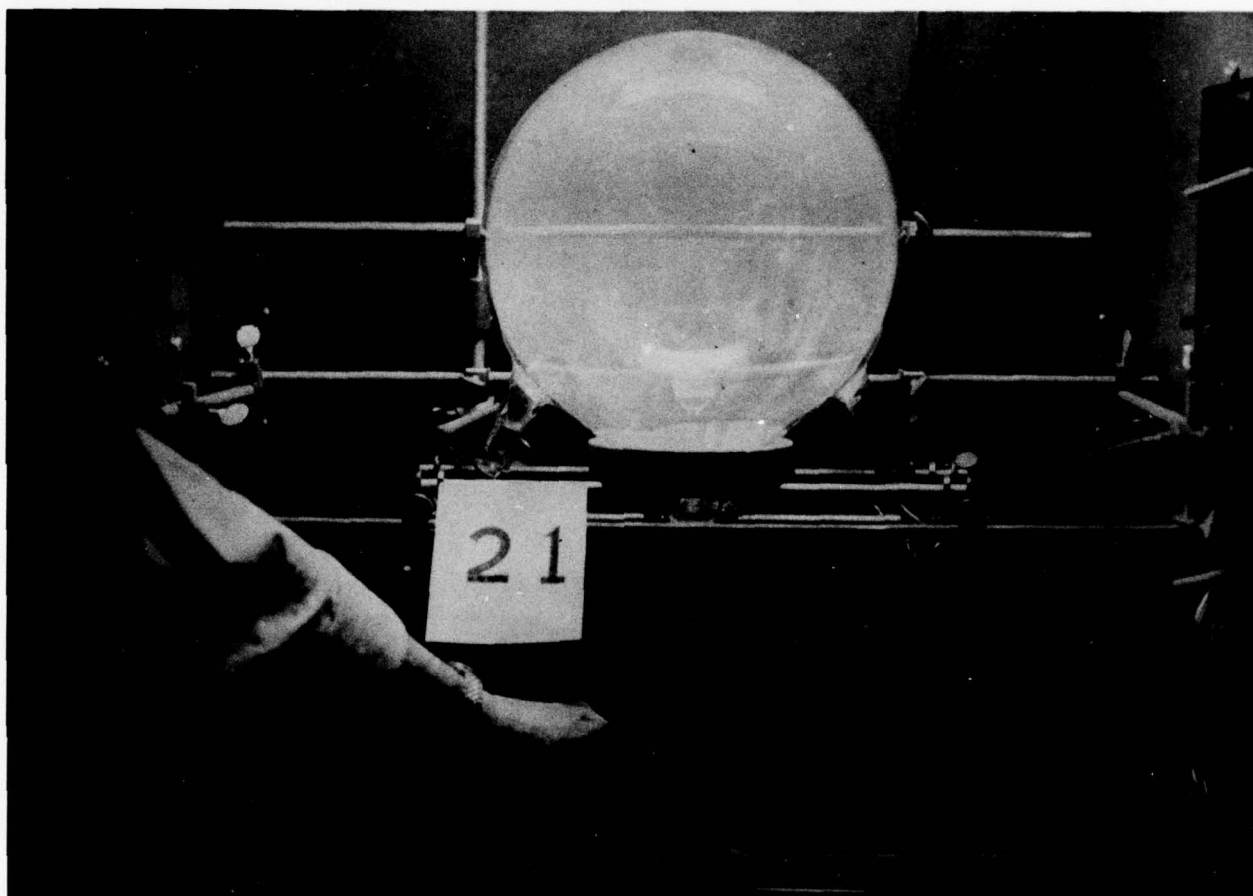


Figure 23. Dispersion of Day-Glo Orange Powder by the Second Method in Experiment 21

E. Smoke Generation from LTSB Mixture I - Experiments 13 and 14

The apparatus employed in experiments 13 and 14 is shown in figure 6. Its use has been described earlier. The composition and method of formulation of LTSB Mixture I have also been given earlier.

The cylindrical sample used in experiment 13 was 1.28 cm in diameter, and 1.40 cm in length. Its volume was  $1.97 \text{ cm}^3$ ; its weight was 2.90 g. A similar sample used in experiment 14 was 1.28 cm in diameter, 1.39 cm in length, had a volume of  $1.94 \text{ cm}^3$ , and weighed 2.82 g.

The results obtained in both of these experiments were very similar. The first appearance of smoke in both cases was nearly 2.5 minutes after placement of the sample furnace over the sample sidearm. Conditions within the flask shortly after the first appearance of smoke are shown in figures 24 and 27 for experiments 13 and 14 respectively. At somewhat later than 3.0 minutes, the conditions in the flask were as shown in figures 25 and 28. The fully developed smokes were achieved at about 3.5 minutes and are shown in figures 26 and 29.

The very first smoke obtained in both cases was white. It appeared suddenly. There then followed a period of about 10 to 20 seconds in which no further accumulation of smoke occurred. Then a sudden rush of additional smoke followed, but from this point on the smoke was light grey. The quantity and quality of the smoke did not equal that obtained in any of the experiments with  $\text{TiCl}_4$ .

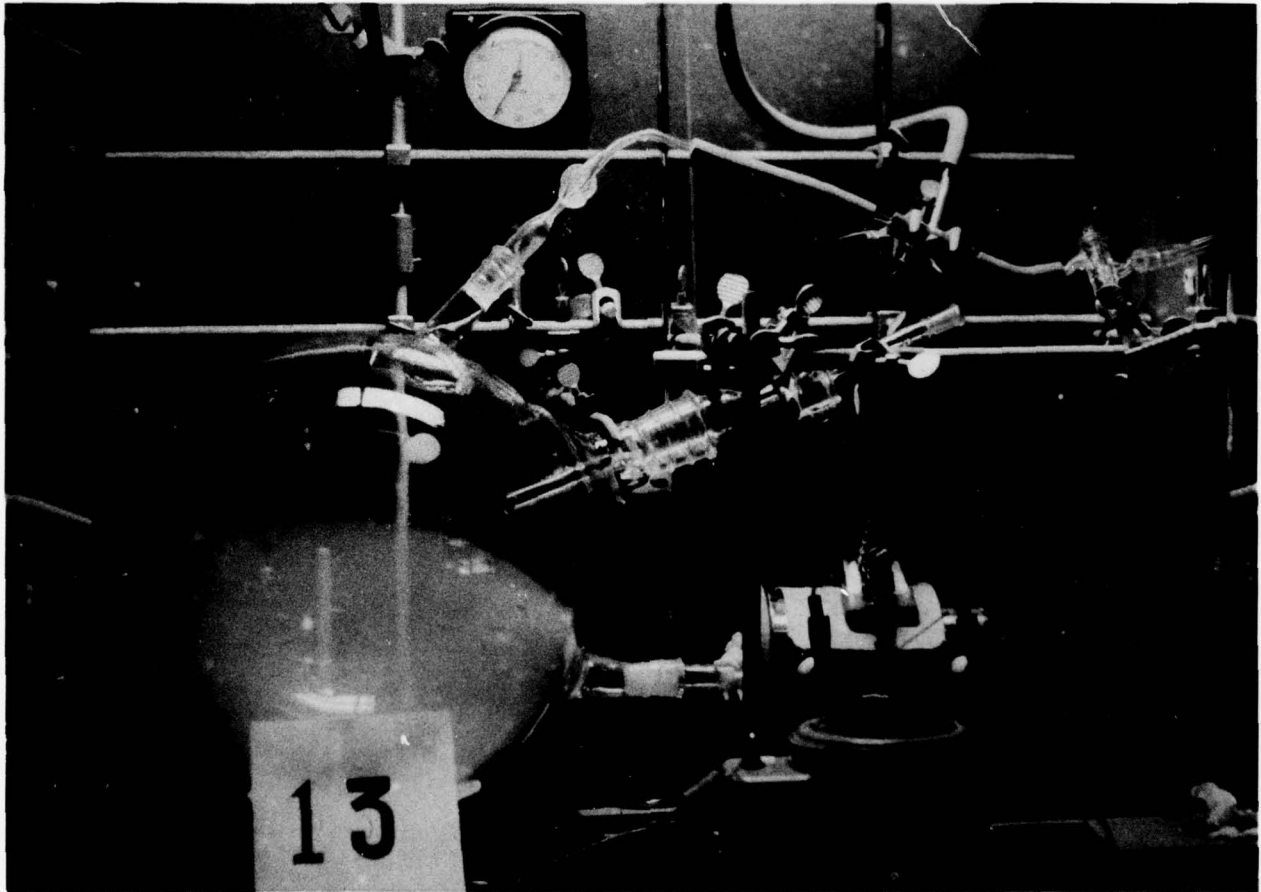


Figure 24. Smoke at an Early Stage of Burning LTSC Mixture I in Experiment 13



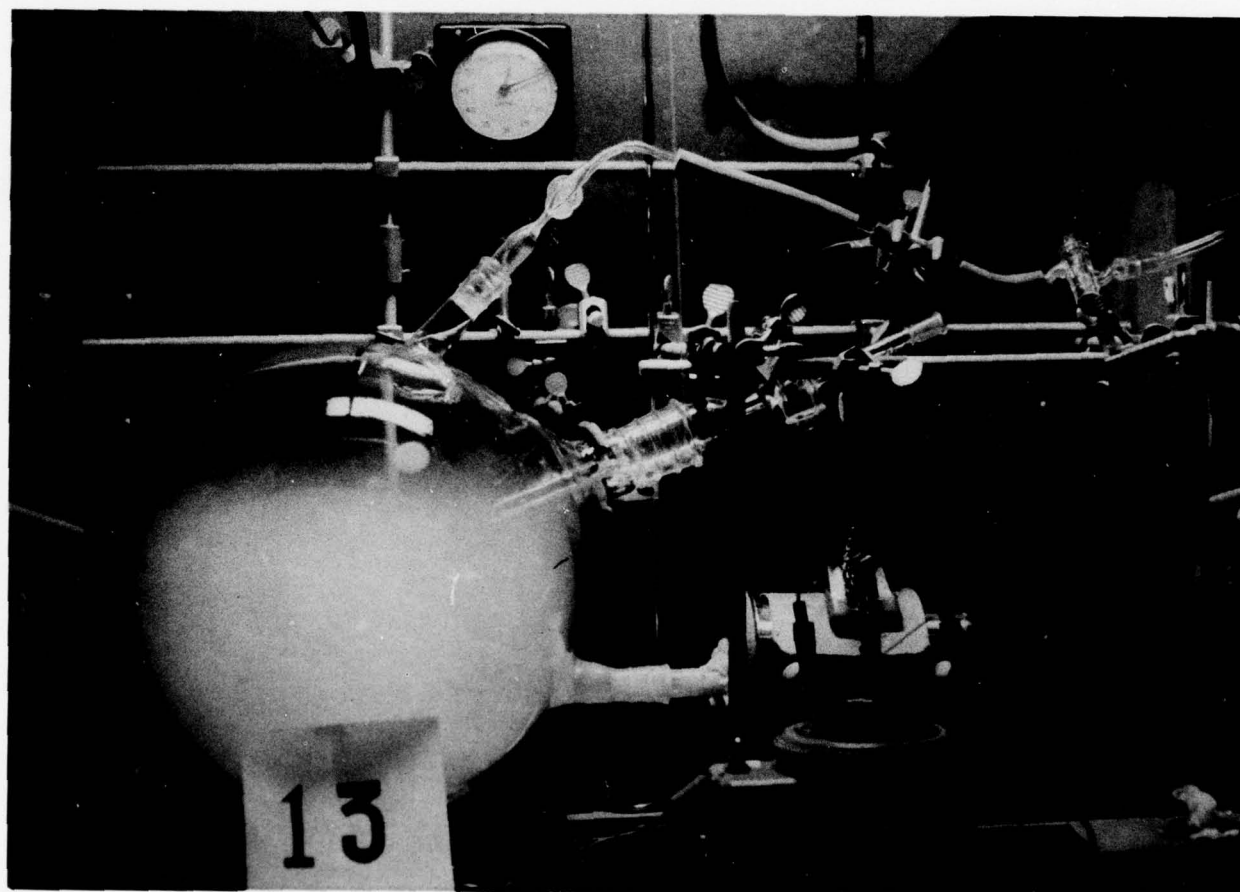


Figure 25. Smoke at a Late Stage of Burning LTSB Mixture I  
in Experiment 13

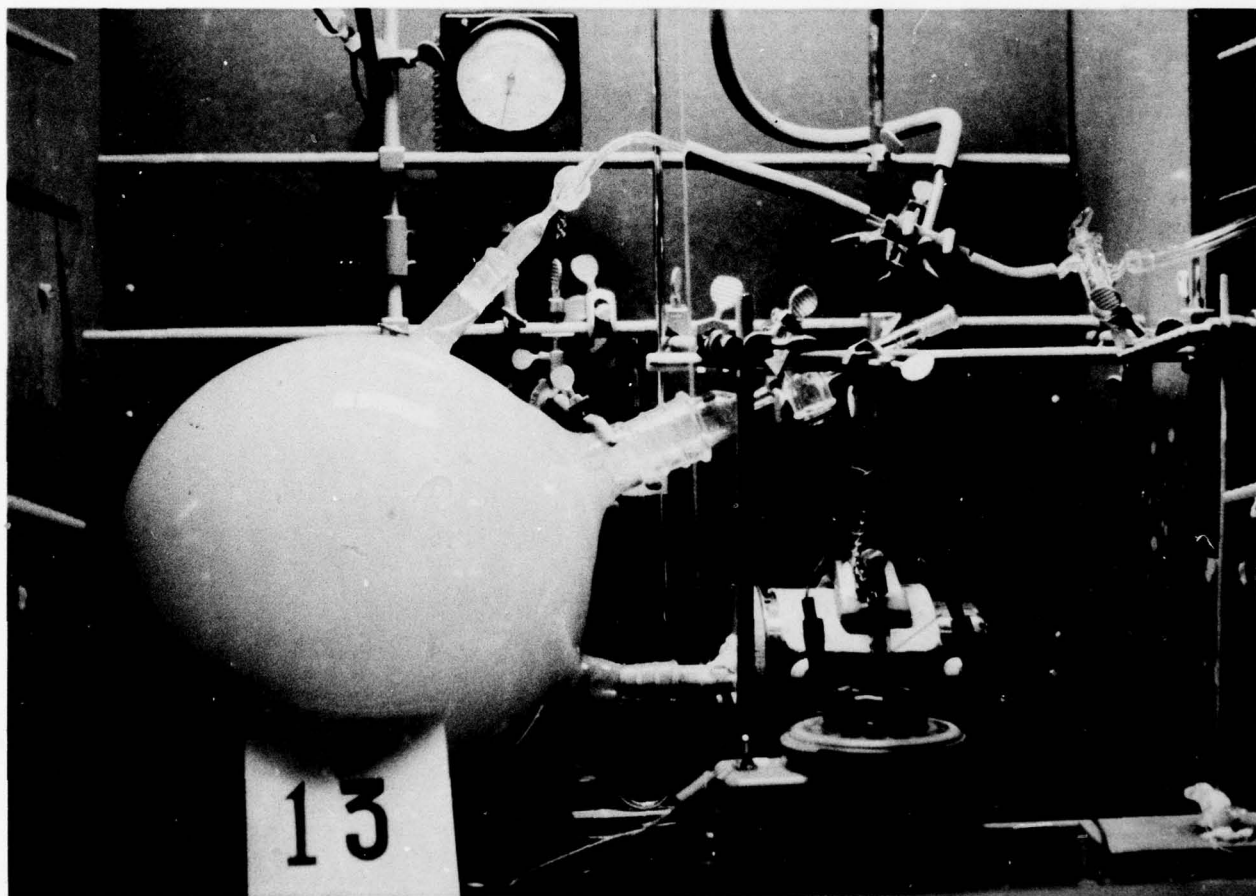


Figure 26. Smoke at Conclusion of Burning LTSE Mixture I in Experiment 13

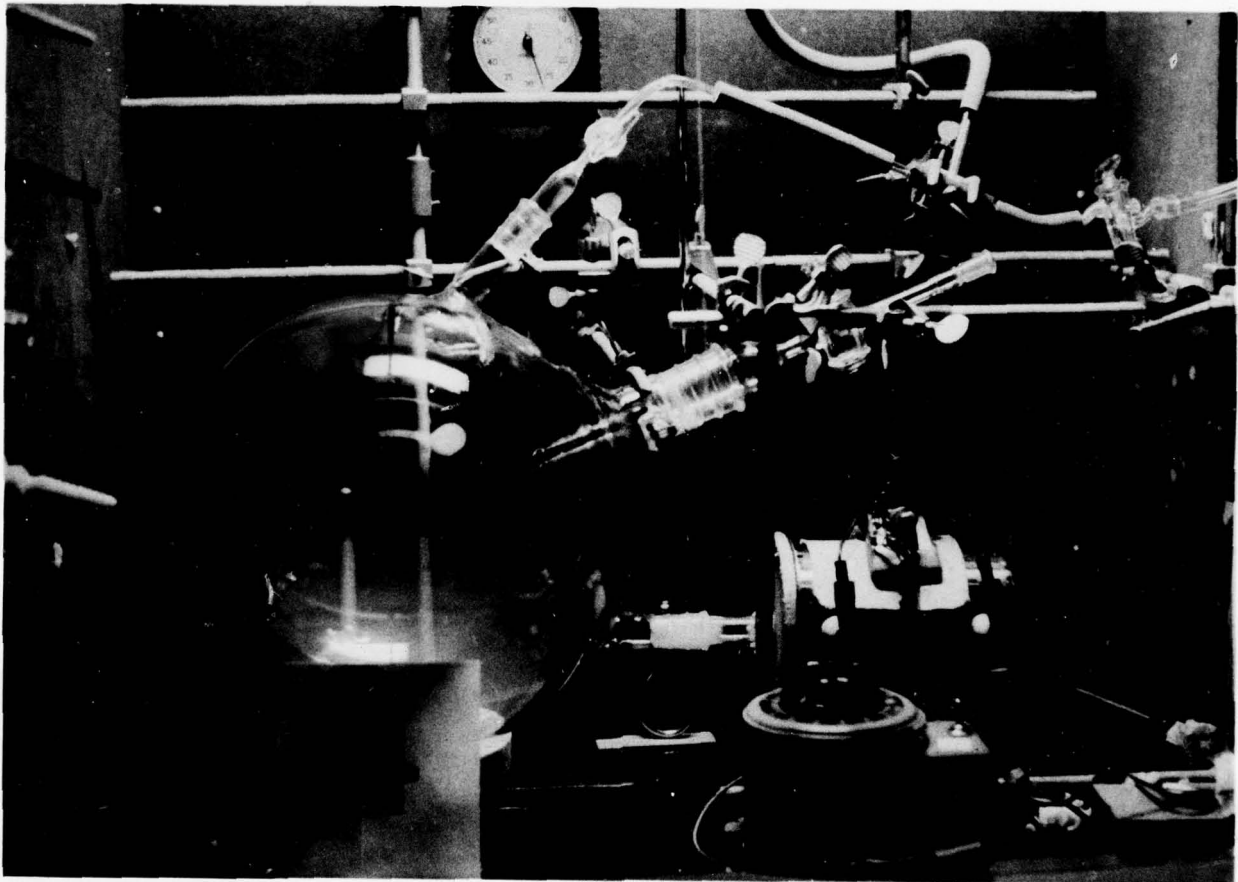


Figure 27. Smoke at an Early Stage of Burning LTSB Mixture I in Experiment 14

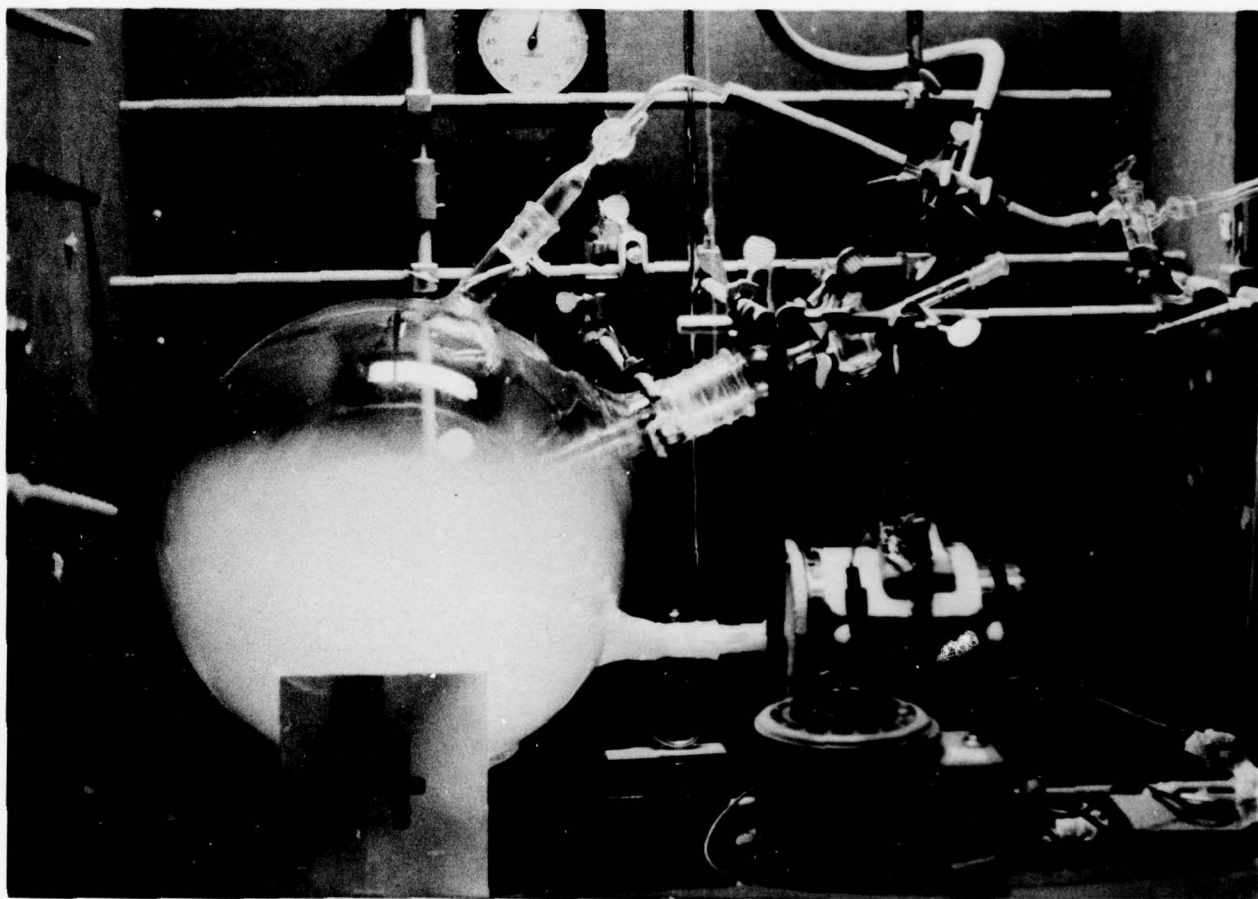


Figure 28. Smoke at a Late Stage of Burning LTSB Mixture I in Experiment 14



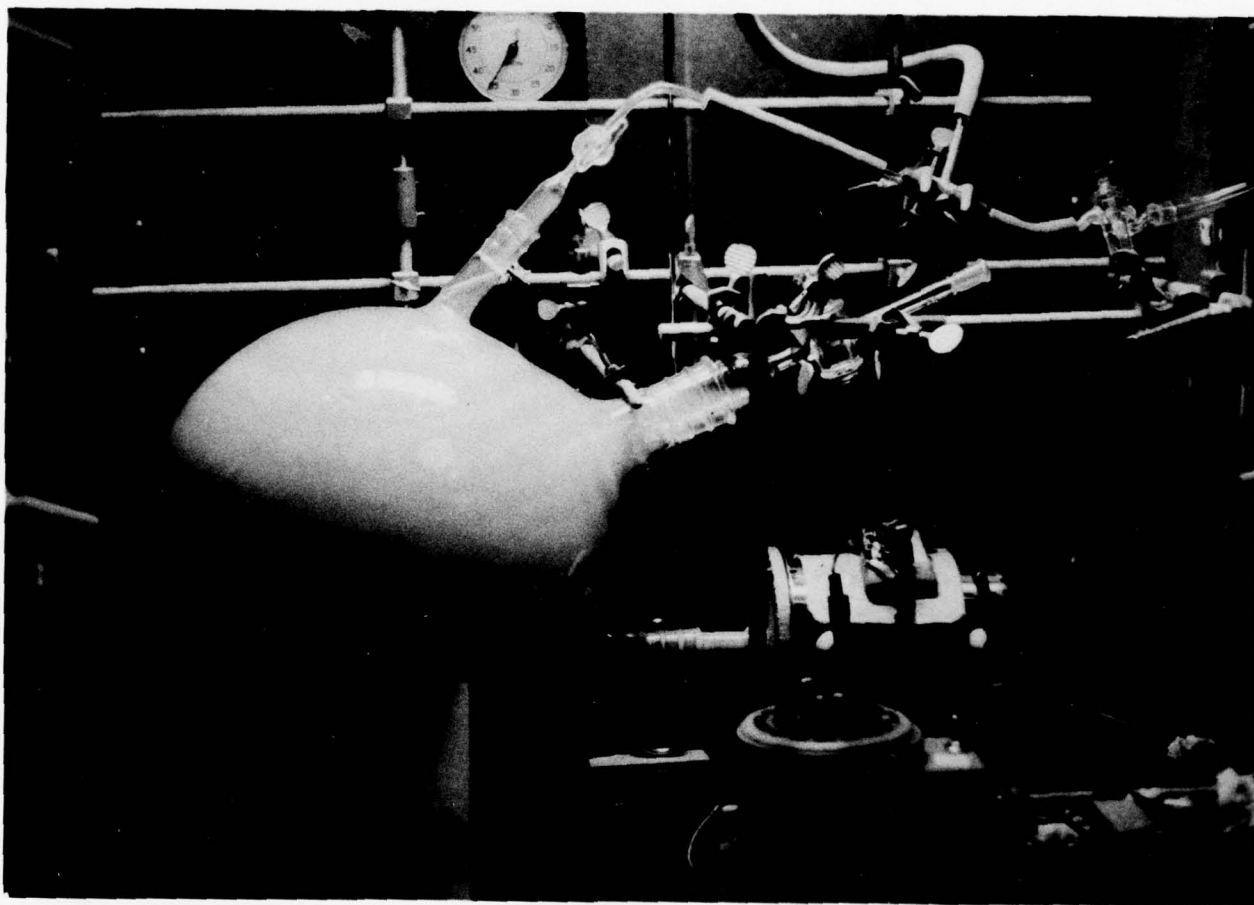


Figure 29. Smoke at Conclusion of Burning LTSB Mixture I in Experiment 14

Examination of the residue in the sample sidearm after each experiment showed that for practical purposes, all of each sample was consumed. Apparently, throughout the consumption of the sample, the pellet remained in its original position and was thereby subjected to the heat from the furnace throughout. These points are made for later comparison with other results.

F. Smoke Generation from LTSB Mixture III - Experiments 15, 16, and 17

The apparatus and procedure employed in these experiments was the same as that used for most LTSB samples. However, in the case of LTSB Mixture III, the sample was a hand-fashioned cylinder of approximately the same size as that of LTSB Mixture I. The soft and pliable nature of Mixture III precluded sample preparation under pressure.

The samples in experiments 15, 16, and 17 weighed 3.72 g, 2.82 g, and 3.19 g, respectively. However, only in experiment 15 was all of the sample consumed. At the end of each of experiments 16 and 17, a substantial lump of hard sample was found at the bottom of the round-bottom flask. The evolution of smoke and gases from these samples apparently propelled a portion of each from the sample sidearm into the cool flask where reaction ceased. The unburned sample particle from experiment 16 weighed 1.47 g, and that from experiment 17 weighed 0.76 g. Therefore, the weights of burned samples were 1.35 g and 2.43 g in experiments 16 and 17, respectively.

The results from these experiments were similar to those with LTSB Mixture I in that there were again two distinct periods of smoke evolution. The smoke in the first period was white, and that in the second was grey. The final smoke mixture was a light greenish-grey.

Figures 30 thru 34 cover these experiments. First smoke came nearly 1.5 minutes after application of heat. Completion of smoke generation required about two minutes. These times are based only on experiment 15 which is the only experiment with LTSB Mixture III in which the sample was completely consumed.

The smoke obtained with LTSB Mixture III was superior to that obtained with LTSB Mixture I, but poorer than any obtained with  $\text{TiCl}_4$ .

G. Smoke Generation from LTSB Mixture II - Experiments 18, 19, and 20

An attempt was made to produce smoke with a pellet of LTSB Mixture II in the same apparatus used with LTSB Mixtures I and III (experiment 18), but no smoke was produced. The pellet diameter was 1.29 cm, and its length was 1.33 cm. Accordingly, its volume was  $1.74 \text{ cm}^3$ , and it weighed 4.25 g.

A preliminary experiment with Mixture II in a porcelain crucible using a hand torch had produced some smoke, but only with difficulty. Accordingly, the apparatus of figure 8 was used in the manner described earlier to attempt to produce smoke from a pellet of Mixture II. Its diameter was 1.29 cm, and its length was 1.28 cm. It had a volume

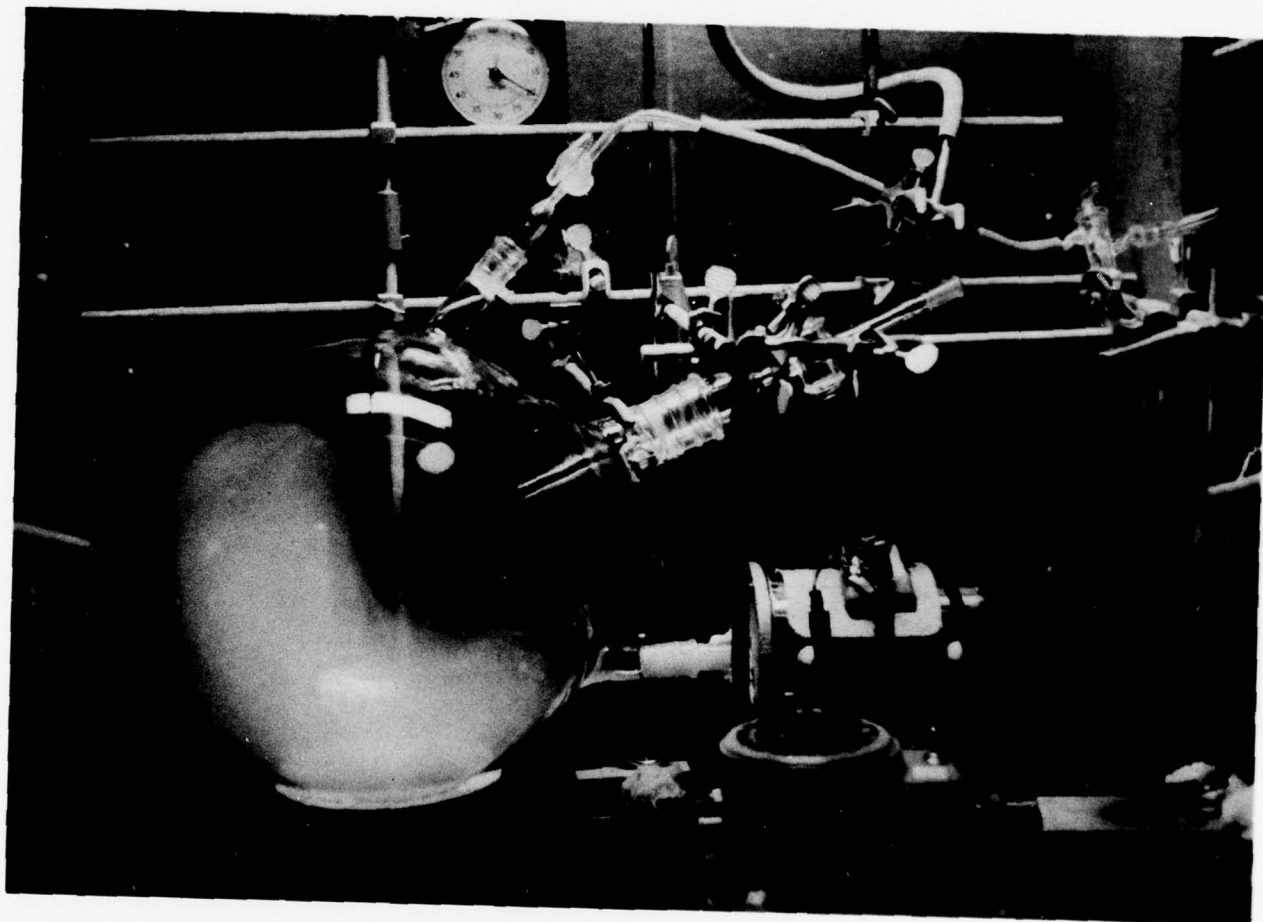


Figure 30. Smoke at an Early Stage of Burning LTSB Mixture III in Experiment 15



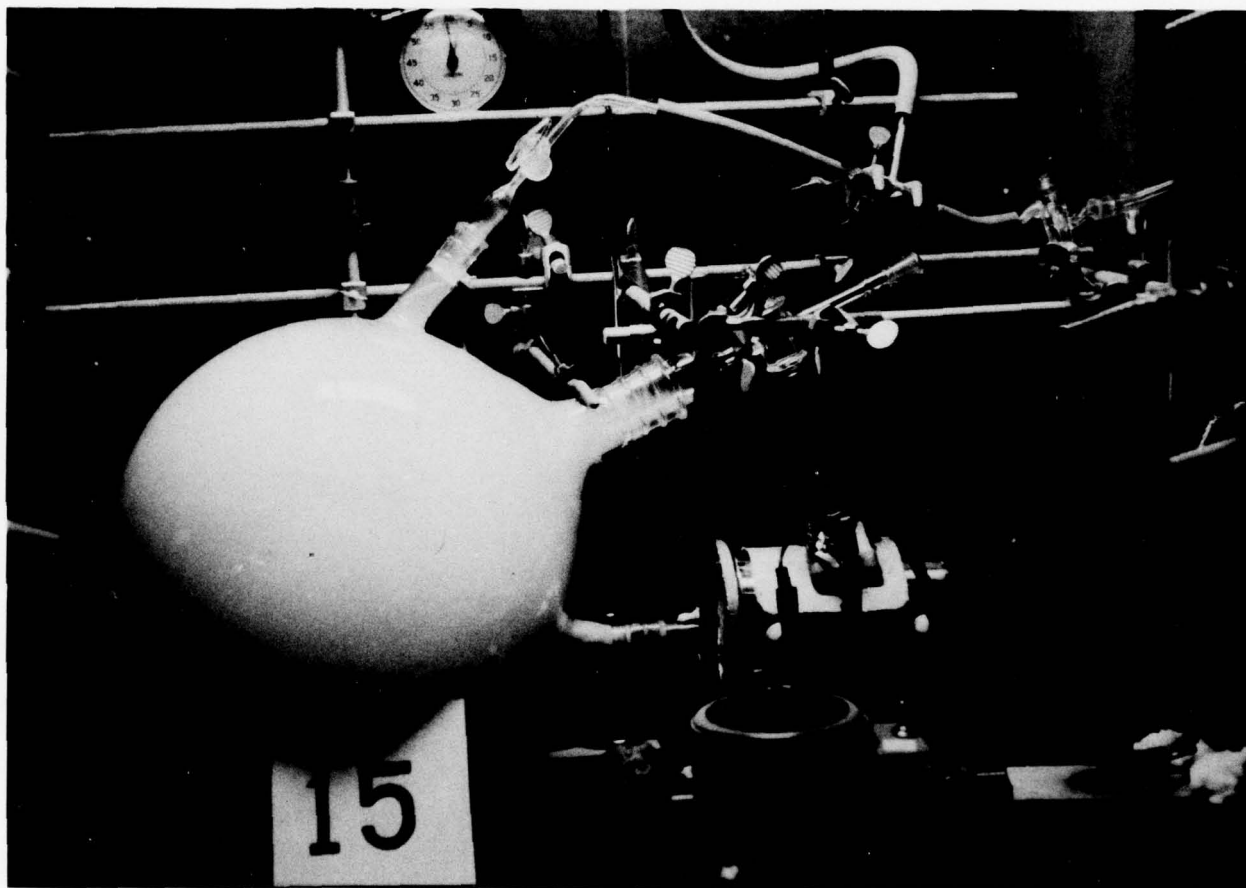


Figure 31. Smoke at Conclusion of Burning LTSE Mixture III in Experiment 15

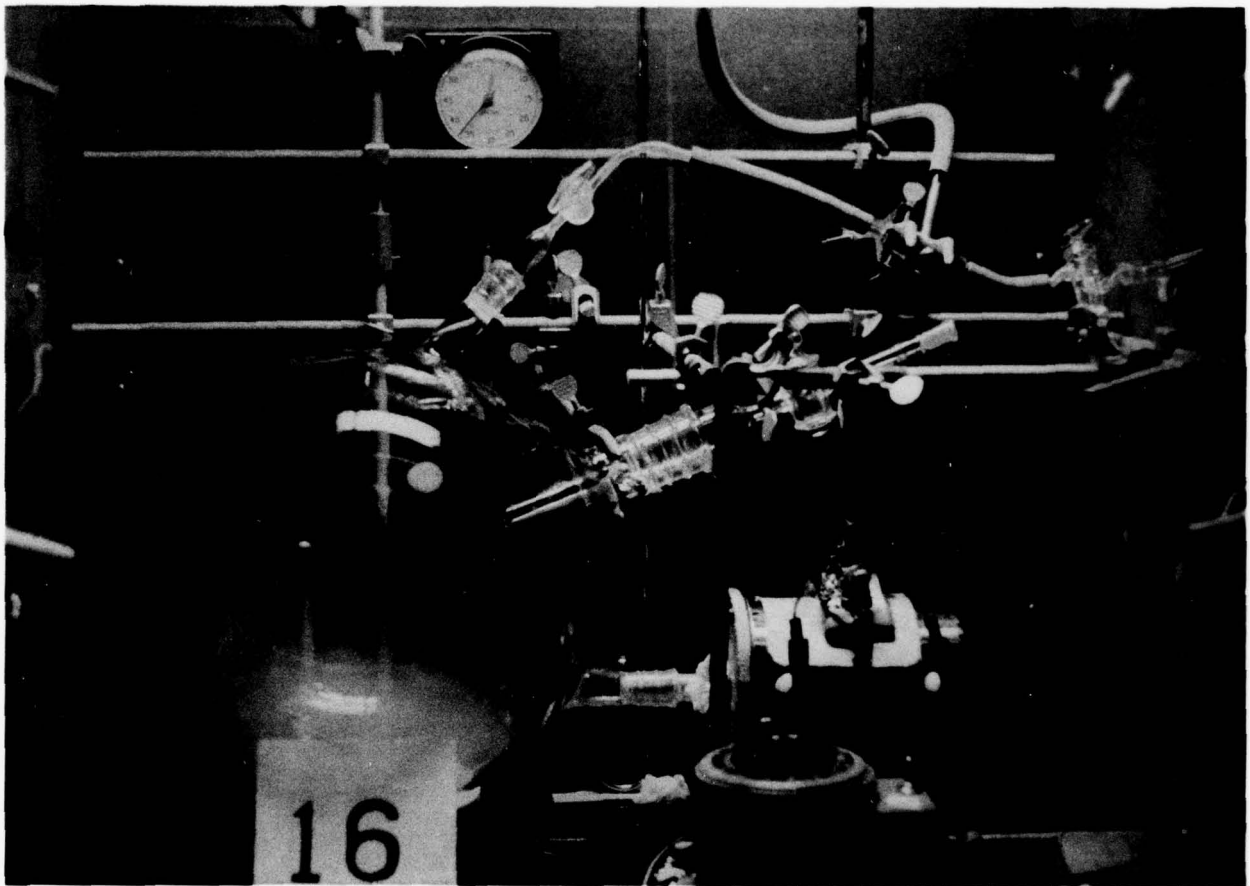


Figure 32. Smoke at an Early Stage of Burning LTSB Mixture III in Experiment 16

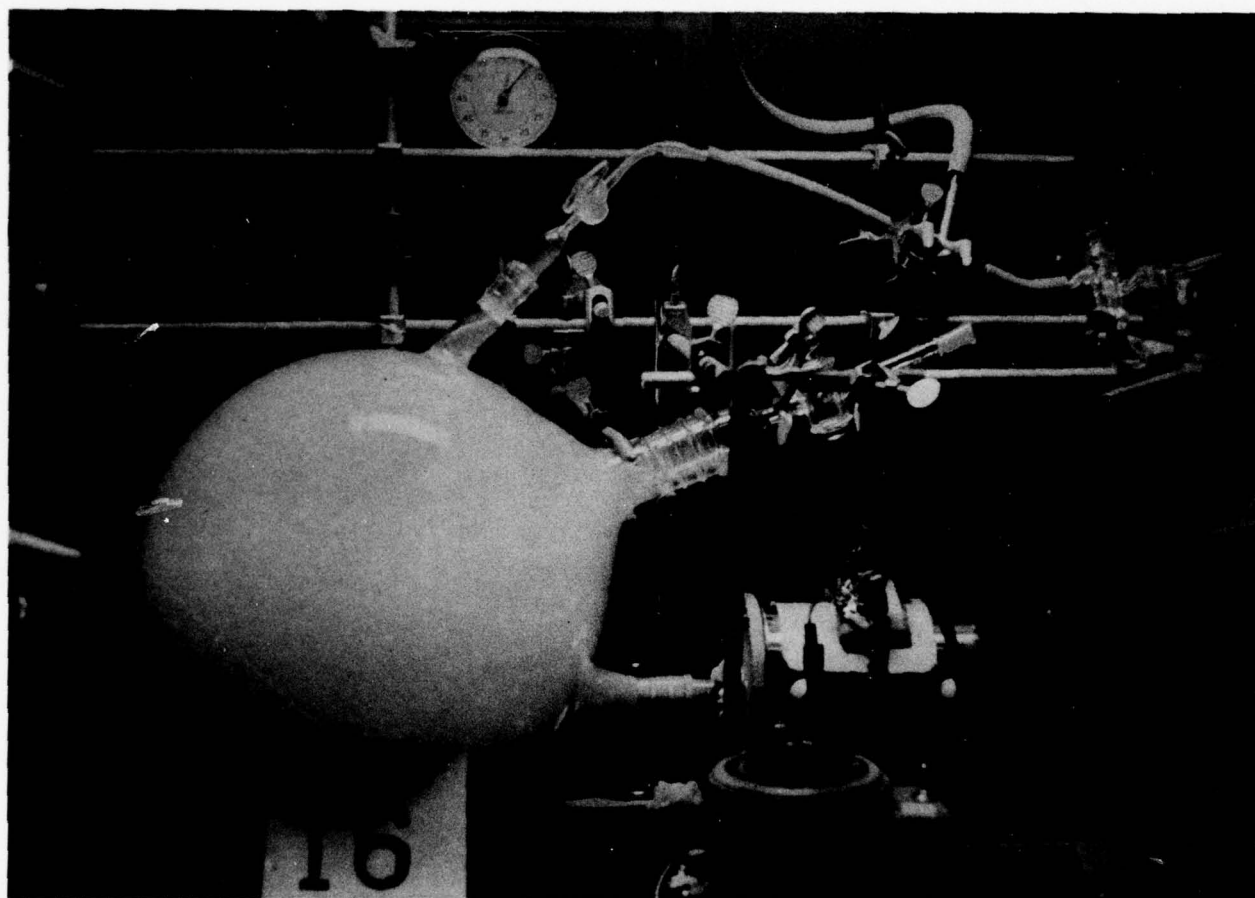


Figure 33. Smoke at Conclusion of Burning LTSB Mixture III in Experiment 16

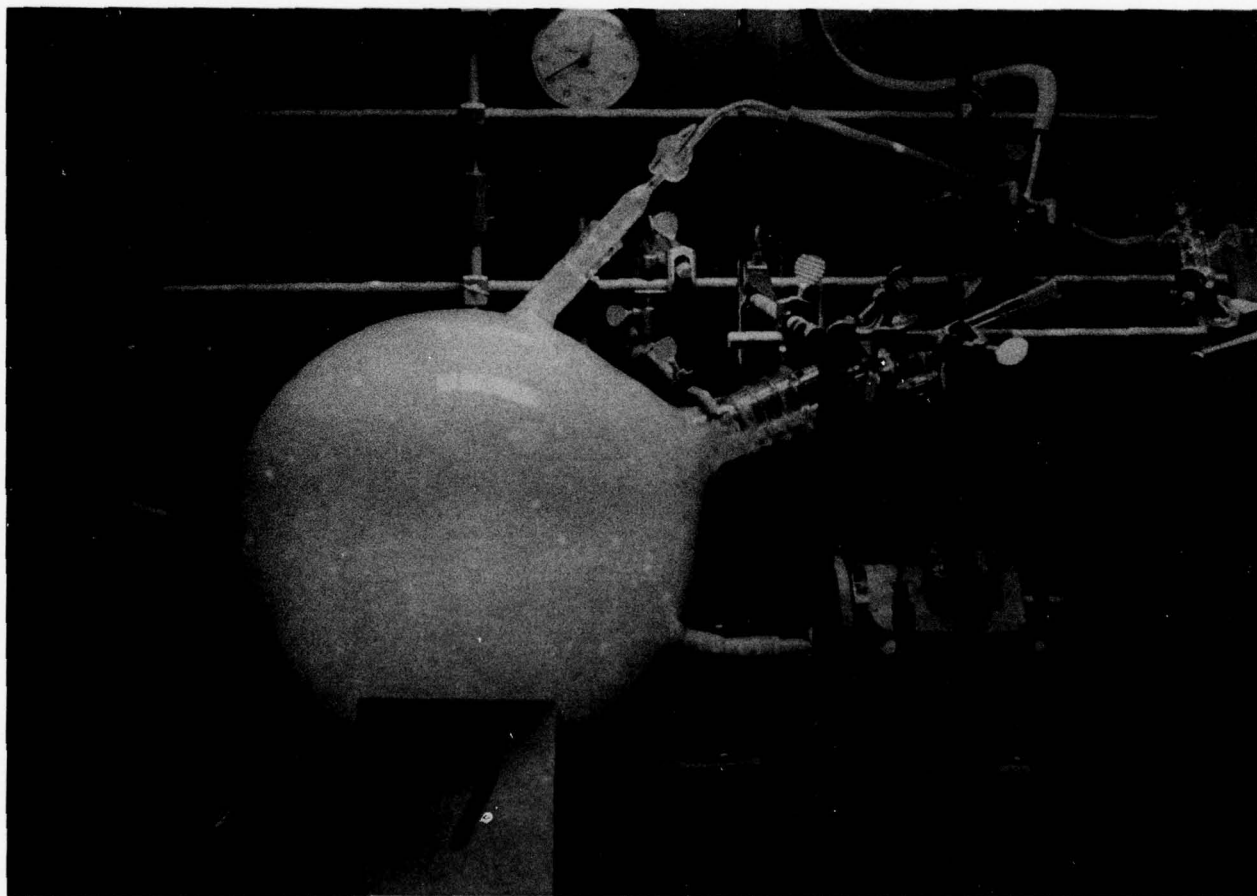


Figure 34. Smoke at Conclusion of Burning LTSB Mixture III in Experiment 17



of  $1.67 \text{ cm}^3$ , and it weighed 4.16 g. The result shown in figure 7 was achieved with great difficulty. The quantity of the smoke was far less than in most of the other experiments. The quality was good in that it was white and was stable, i.e., it did not settle quickly.

In the last experiment with Mixture II (experiment 20), 4.16 g of uncompact powder was treated in the same manner as the sample in experiment 19. The result is shown in figure 35.

There was nothing that distinguished the smoke of experiment 20 from that of experiment 19. What burning did occur in experiment 20 took place in a shorter period of time than that of experiment 19, but in experiment 20, the sample was in better contact with the heated crucible than was the sample of experiment 19. In both of these cases, most of the sample remained unburned.

##### 5. Comments on the LTSB Mixtures

With the spontaneously reacting chemicals, reaction takes place instantly at ambient temperature when the chemicals are mixed either as liquids or as gasses. On the other hand, the LTSB mixtures are more complex. These mixtures have a relatively large number of formulation variables and material properties upon which successful and satisfactory generation of a smoke depend when these mixtures are heated. It is unlikely that the LTSB mixtures used in this study were formulated in the best manner, or that the materials used possessed those properties (such as particle size) which optimized these particular chemicals for their intended purpose.

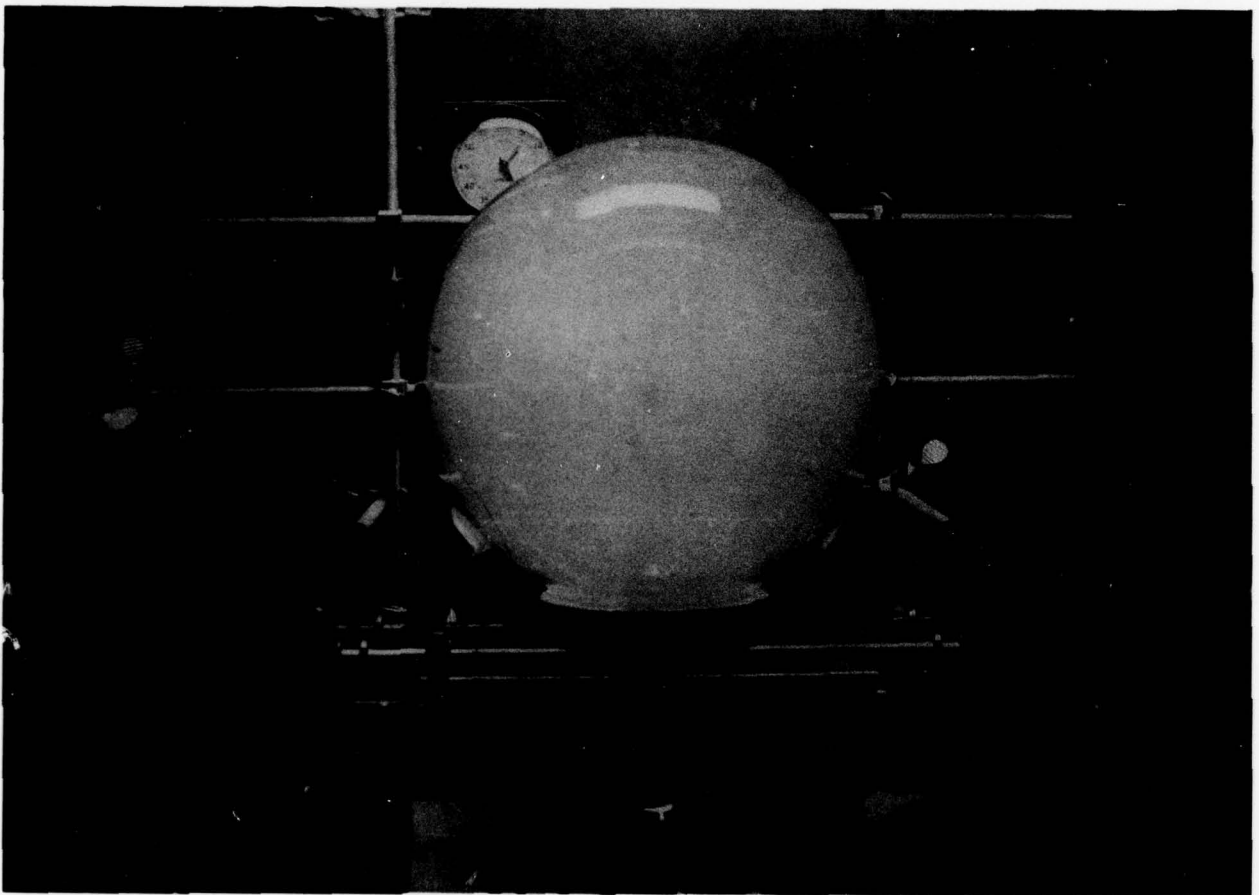


Figure 35. Smoke at Conclusion of Burning LTSB Mixture II in Experiment 20

The LTSB mixture containing hexachloroethane offers an example of a mixture that has performed at times in an unsatisfactory and non-reproducible manner when used by others. By careful materials management and control of formulation procedures, it has been possible to improve the performance of this mixture. See Reference 1.

To the knowledge of these authors, the influence of microcrystal structure of zinc oxide, of nonstoichiometry of zinc oxide, and of surface impurities and surface structure of aluminum powder, on the reactions of burning LTSB Mixture II, have never been studied.

#### 6. Properties of Chemicals Used

This section of the report is a listing of the properties of the chemicals used in the experiments described herein. These properties should be considered when handling these substances.

Although acetyl chloride was used in some experiments, it is not one of the materials being considered for smoke generation, and is accordingly not included in the list below. Others not included below are those for which nothing could be found in the reference consulted.

The information presented here has been abstracted from Reference 2 as seemed most appropriate.



a. Aluminum

1. Toxic Hazard Ratings:

Acute local: None  
Acute systemic: None  
Chronic local: Inhalation-slight  
Chronic systemic: None

2. Toxicology:

Some dispute. Generally not regarded as an industrial poison. Some recorded instances (2) of acute lung fibrosis in manufacture and use of the powder.

b. Aluminum Dust

1. Toxic Hazard Ratings:

Acute local: Irritant-slight;  
Inhalation-slight  
Acute systemic: None  
Chronic local: Inhalation-slight  
Chronic systemic: None

2. Toxicology:

Can irritate eyes. European reports of chronic pulmonary disease due to inhalation.

c. Ammonium Hydroxide

1. Toxic Hazard Rating:

Acute local: Irritant-moderate;  
Ingestion-high;  
Inhalation-moderate  
Acute systemic: Unknown  
Chronic local: Irritant-moderate  
Chronic systemic: Unknown

2. Toxicology:

Not recorded.



d. Hexachloroethane

1. Toxic Hazard Rating:

Acute local: Irritant-moderate;  
Ingestion-moderate  
Acute systemic: Ingestion-moderate  
Chronic local: Unknown  
Chronic systemic: Ingestion-moderate;  
Inhalation-moderate

2. Toxicology:

Not recorded.

e. Potassium Chlorate (Same for all chlorates)

1. Toxic Hazard Rating:

Acute local: Irritant-slight  
Acute systemic: Ingestion-moderate  
Chronic local: Irritant-slight  
Chronic systemic: Ingestion-moderate

2. Toxicology:

Principal toxic effects are production of methemoglobin in the blood and destruction of red blood corpuscles. Result may be irritation of kidneys. Heart damage reported!

f. Sulfur, flowers of.

Toxicity: Very low. A nuisance.

g. Sulfuryl Chloride

This substance reacts rapidly with water and water-containing systems producing hydrochloric and sulfuric acids. Both of these have the following ratings.

1. Toxic Hazard Ratings:

Acute local: Irritant-high;  
Ingestion-high;  
Inhalation-high  
Acute systemic: Unknown  
Chronic local: Irritant-moderate  
Chronic systemic: Unknown

## 2. Toxicology:

Hydrochloric acid is an irritant to mucous membranes. The severity of reaction to it depends on the exposure. Sulfuric acid destroys tissue causing severe burns. Both are considered as dangerous materials.

### h. Titanium Tetrachloride

This is an irritant material and is also corrosive because of generation of hydrochloric acid when in contact with moisture or moisture containing systems. Hazard is reduced considerably if hydrochloric acid is neutralized by the use of ammonium hydroxide as a reactant.

### i. Zinc Oxide

Generally low toxicity. Virtually having no ill effects.

## RECOMMENDATIONS

On the basis of the observations of the smokes produced, their quantities, qualities, and ease of production, the following systems appear to be suitable for preparation of mortar smoke rounds for field tests. These are listed in order of decreasing preference.

1. Titanium tetrachloride and ammonium hydroxide.
2. Sulfuryl chloride and ammonium hydroxide.
3. LTSB Mixture III.

The first system recommended is based upon the results obtained with  $\text{TiCl}_4$  and water, and the determination that the use of ammonium hydroxide (28-percent by weight of aqueous ammonia) gives equally desirable results, and appreciably reduces toxicity problem. The  $\text{TiCl}_4\text{-NH}_4\text{OH}$  experiment for which the calculations have been included in this report is described in a supplement to this document.

Among the reasons for recommending the first system is that titanium dioxide and hydrous titanium oxide are not harmful. The ammonium chloride expected in the smoke presents only a slight toxic hazard (Reference 2). The important requirement here is that the real mortar round achieve thorough mixing of the chemicals to achieve conversion of all hydrogen chloride (hydrochloric acid) to  $\text{NH}_4\text{Cl}$ . An excess of  $\text{NH}_4\text{OH}$  can be used to increase the probability of this effect.

The same argument can be made about the  $\text{NH}_4\text{Cl}$  and  $(\text{NH}_4)_2\text{SO}_4$  products from the reaction of  $\text{SO}_2\text{Cl}_2$  and  $\text{NH}_4\text{OH}$  (second system).



## DESIGN AND COST

A mortar fuze mechanical design to implement the binary system is shown in figure 36.

A cost estimate for large scale productions of the fuze, with explanatory breakdown follows figure 36.



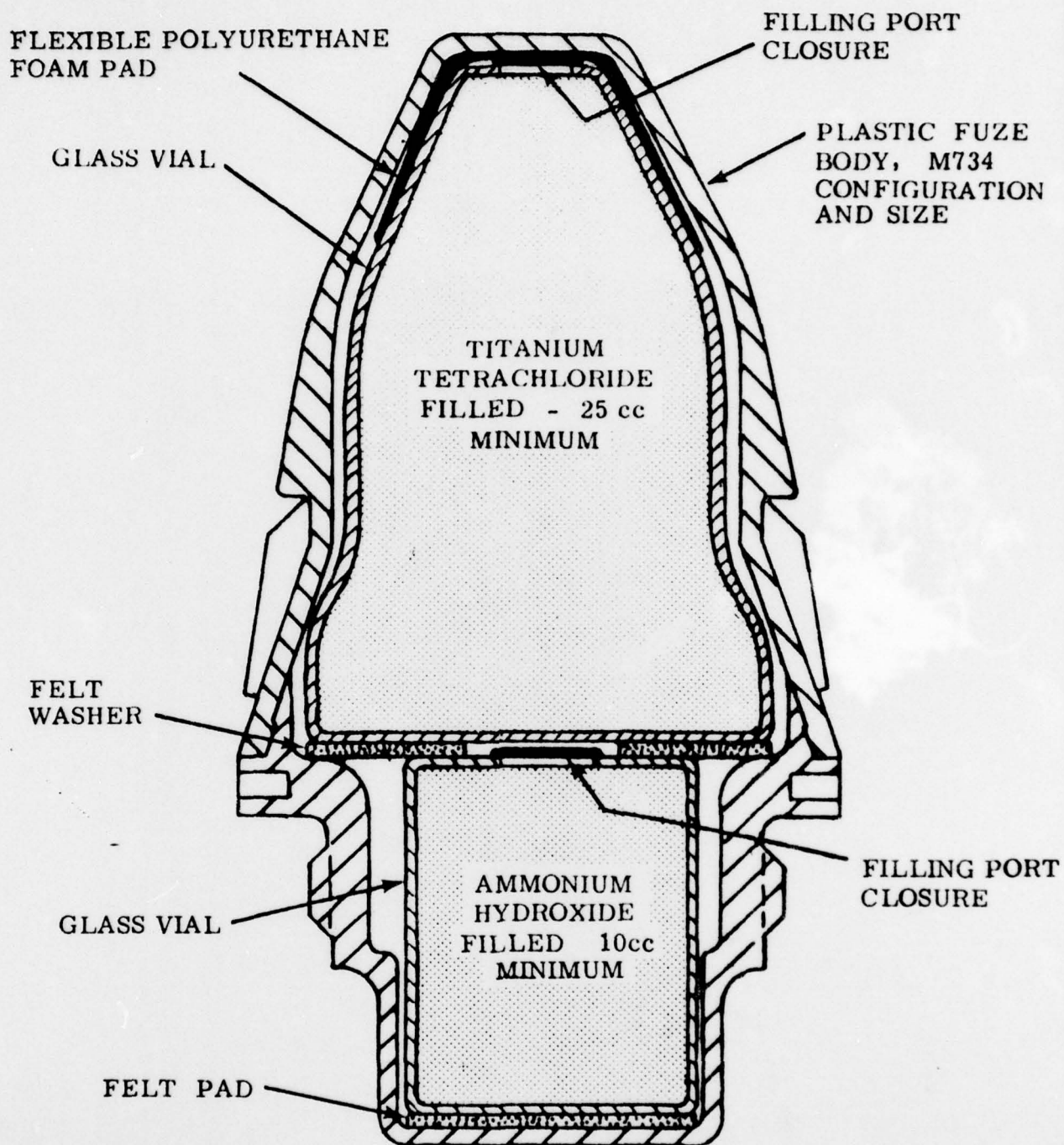


Figure 36. Practice Fuze, 60/81-mm Mortar Hazard-Free, Binary Chemical Smoke

**BEI Electronics Inc.**  
**Defense Products Division**

11312 S. Pipeline Rd. Euless, Texas 76039

COST ESTIMATE\*

Practice Fuze, Hazard Free, Binary Chemical

500,000 Units Per Year

A. Recurring Costs (Note 1)

(1)	Production Labor (Attachment I)	--	\$ .231
(2)	Factory Overhead @ 150%	--	.347
(3)	Component and Materials (Attachment II)	--	<u>\$1.490</u>
	TOTAL FACTORY COST	--	\$2.068 each

B. Non-Recurring Costs (Note 1)

(1)	Preparation of Program Quality Plan, Manufacturing Process Instructions, and related Documentation	--	\$ 4,000.00
(2)	Vendor Tooling (Attachment II)		4,250.00
(3)	Production Tooling and Facility Set-up, (Attachment III)		<u>12,500.00</u>
	TOTAL FACTORY COST	--	\$20,750.00

\* All Costs are in 1st Qtr - 1979 Dollars

Note I G&A Expense and Profit Allowance Not Included

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**Defense Products Division**

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ATTACHMENT I

Practice Fuze, Hazard Free, Binary Chemical

500,000/Yr - 41,700/Mo. - 2,000/1 Shift Day

Production Rate - 5/Min. - 400 Min. Shift

<u>LABOR *</u>			
<u>TASK</u>	<u>MANNING</u>	<u>RATE</u>	<u>\$/SHIFT</u>
1. Supervision	1 Foreman	\$7.75	\$ 62.00
2. Inspection	1 Inspector	3.94	31.52
3. Material Handling	1 Assembly Worker	3.84	30.72
4. Purge Glass Vials with N <sub>2</sub>	2 Assembly Workers	3.84	61.44
5. Fill TiCl <sub>4</sub> Vial	2 Assembly Workers	3.84	61.44
6. Fill NH <sub>4</sub> OH Vial	2 Assembly Workers	3.84	61.44
7. Mix Epoxy and apply Closure Disc to Vials	3 Assembly Workers	3.84	92.16
8. Final Assembly	2 Assembly Workers	3.84	61.44
TOTAL	14		\$462.16

\* Packaging Not Included

2,000 Unit - 112 MH - \$462.16

Unit - .056 MH/E - \$ .231 each



ATTACHMENT II

BEI ELECTRONICS, INC.  
1101 McAlmont Street  
Little Rock, Ark. 72203

**BILL OF MATERIAL**

PAGE 1 of 1

Schedule:

PROGRAM: Practice Fuze, Hazard Free, Smoke			TOTAL QUANTITY REQUIRED	NON- RECURRING COST	FREIGHT COST	ITEM UNIT COST	EXTENDED COST	REMARKS
DESCRIPTION	PART NO. DWG.	PROSPECTIVE SUPPLIER						
1. Fuze Body, Plastic Filled Nylon	40% Glass	Engineered Plastics Philadelphia, Pa.	510,000	Tooling Existing		\$ .900	\$459,000	
2. Glass Vial, Nose Piece		Franklin Glass Co.	515,000	2,400		.171	88,065	
3. Glass Vial, Base Piece		Franklin Glass Co.	515,000	1,400		.136	70,040	
4. Felt Pad, Base		-	510,000	100		.015	7,650	Eng. Est.
5. Felt Washer		-	510,000	350		.030	15,300	Eng. Est.
6. Filling Port Closures		-	1,025,000	--		.020	20,500	Eng. Est.
7. Epoxy, Two Part	#1838 B/A 3M		41 Qts.			25.000	1,025	
8. Titanium Tetrachloride		Stauffer Chem.	52,000#			.77	40,040	
9. Ammonium Hydroxide, Aqueous		Allied Chemical	18,200#			.23	4,186	
10. Misc. Material Nitrogen, Etc.		-	510,000			.05	25,500	
SUBTOTAL							\$731,306	
11. Incoming Freight - 2%							14,626	
TOTAL COST				\$4,250			\$745,932	
UNIT COST							\$1.49	
Packaging Material - Not included.								



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**Defense Products Division**

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ATTACHMENT III

Practice Fuze, Hazard Free, Binary Chemical

500,000/Yr - 41,700/Mo. - 2,000/1 Shift Day

Production Rate - 5/Min. - 400 Min. Shift

PRODUCTION TOOLING, FACILITY SET-UP

- NON-RECURRING -

1. Set up Nitrogen Purging Station	--	\$ 2,000
2. Set up $TiCl_4$ Filling Station	--	4,500
3. Set up $NH_4OH$ Filling Station	--	1,500
4. Set up Epoxy Mixing Closure Stations	--	1,500
5. Molding Fixture and Racks	--	2,000
6. Miscellaneous Tools and Fixtures	--	<u>1,000</u>
		\$12,500

#### REFERENCES

1. Defense Documentation Center, Alexandria, Virginia, Document No. AD-B027983-L, Technical Report No. 17, "HC Mix Product Improvement Study", November, 1977, by Wendell L. Fortner, Steven L. Yelldell, and Thomas E. Shook.
2. Sax, N. Irving, "Dangerous Properties of Industrial Materials", Reinhold Publishing Corporation, New York, 1957.



DEPARTMENT OF THE ARMY  
US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND  
DOVER, NEW JERSEY 07801

*Mr. Bill Marks*  
A. Shane/men/201-328-6378

DRDAR-LCN-T

APR 17 1979

BEI Electronics  
Defense Products Division  
Fort Worth, Texas  
ATTN: Mr. E. Snoyer

Dear Mr. Snoyer,

As agreed upon during today's telephone conversation, I am returning the manuscript copy of the final report for Contract DAAK10-78-C-0077. The detailed cost estimate which was omitted from the manuscript copy and which you forwarded later is also being returned. These items are being sent to Mr. Bill Marks, at your Little Rock Office. The report, incorporating the cost estimate, is approved for publication without further revision.

The cartridge designs proposed for demonstration samples, using titanium tetrachloride/ammonium hydroxide and titanium tetrachloride only, are also acceptable.

Copy furnished:  
✓ Mr. Bill Marks  
BEI Electronics Inc  
1101 McAlmont St.  
Little Rock Ark 72203

S. H. ISRAELS  
C, Integral Fuzing Sec.  
Tube Fired Fuze Br.  
Nuclear & Fuze Div, LCWSL